Lower Passaic River Restoration Project

Final Quality Assurance Project Plan

August 2005

PREPARED BY:
Malcolm Pirnie, Inc.
104 Corporate Park Drive
White Plains, NY 10602

FOR:
US Environmental Protection Agency
Region II

US Army Corps of Engineers
Kansas City District
Contract No.
DACW41-02-D-0003

LOWER PASSAIC RIVER RESTORATION PROJECT
FINAL QUALITY ASSURANCE PROJECT PLAN

U.S. ARMY CORPS OF ENGINEERS CONTRACT NO. DACW41-02-D-0003

CONTRACTOR QC SIGN-OFF

Malcolm Pirnie, Inc. has reviewed this document in accordance with the Final Quality Control Plan for the Lower Passaic River Restoration Project, July 2003.

Len Warner
Project Manager

Date: 8/29/05

Bruce Fidler, P.E.
Technical Director

Date: 8/25/05

Jim McCann
Site Quality Control Officer

Date: 8/29/05

QUALITY ASSURANCE PROJECT PLAN

LOWER PASSAIC RIVER RESTORATION PROJECT

Prepared by:

Malcolm Pirnie, Inc., in conjunction with Battelle and HydroQual, Inc.

August 2005

Version: 2005/08/26

TABLE OF CONTENTS

FIGURES

TABLES

ATTACHMENTS

1.0 PROJECT MANAGEMENT

This Quality Assurance Project Plan (QAPP) is developed to address the quality assurance and quality control (QA/QC) elements for the Lower Passaic River Restoration Project. It details the planning processes for collecting data and describes the implementation of the QA and QC activities developed for this program. The purpose of this QAPP is to generate project data that are technically valid and legally defensible.

The QAPP consists of four main components:

- Project Management.
- Measurement and Data Acquisition.
- Assessment and Oversight.
- Data Validation and Usability.

The above components will incorporate QA/QC requirements cited within the following documents:

- U.S. Environmental Protection Agency (USEPA) Requirements for Quality Assurance Project Plans, USEPA QA/R-5, March 2001.
- USEPA Guidance for Quality Assurance Project Plans, USEPA QA/G-5, December 2002.
- USEPA Guidance for the Data Quality Objectives Process, QA/G-4, August 2000.

1.1 DISTRIBUTION LIST

A hard copy of the QAPP will be distributed to the following:

- USEPA, Region 2: Emergency and Remedial Response Division (ERRD) and Division of Environmental Science and Assessment (DESA).
- U.S. Army Corps of Engineers – Kansas City District (USACE-KC).
- U.S. Army Corps of Engineers – New York District (USACE-NY).
- New Jersey Department of Environmental Protection (NJDEP).
- United States Fish and Wildlife Service (USFWS).

- New Jersey Department of Transportation, Office of Maritime Resources (NJDOT-OMR).
- National Oceanic and Atmospheric Administration (NOAA)

An electronic copy of the QAPP will be posted on the Passaic River Estuary Management Information System (PREmis), an internal project website. PREmis is further described in Section 2.10 – Data Management. Using PREmis the project team and individuals associated with the project can access the latest version of this document. The final QAPP will also be posted on the public website, www.ourPassaic.org.

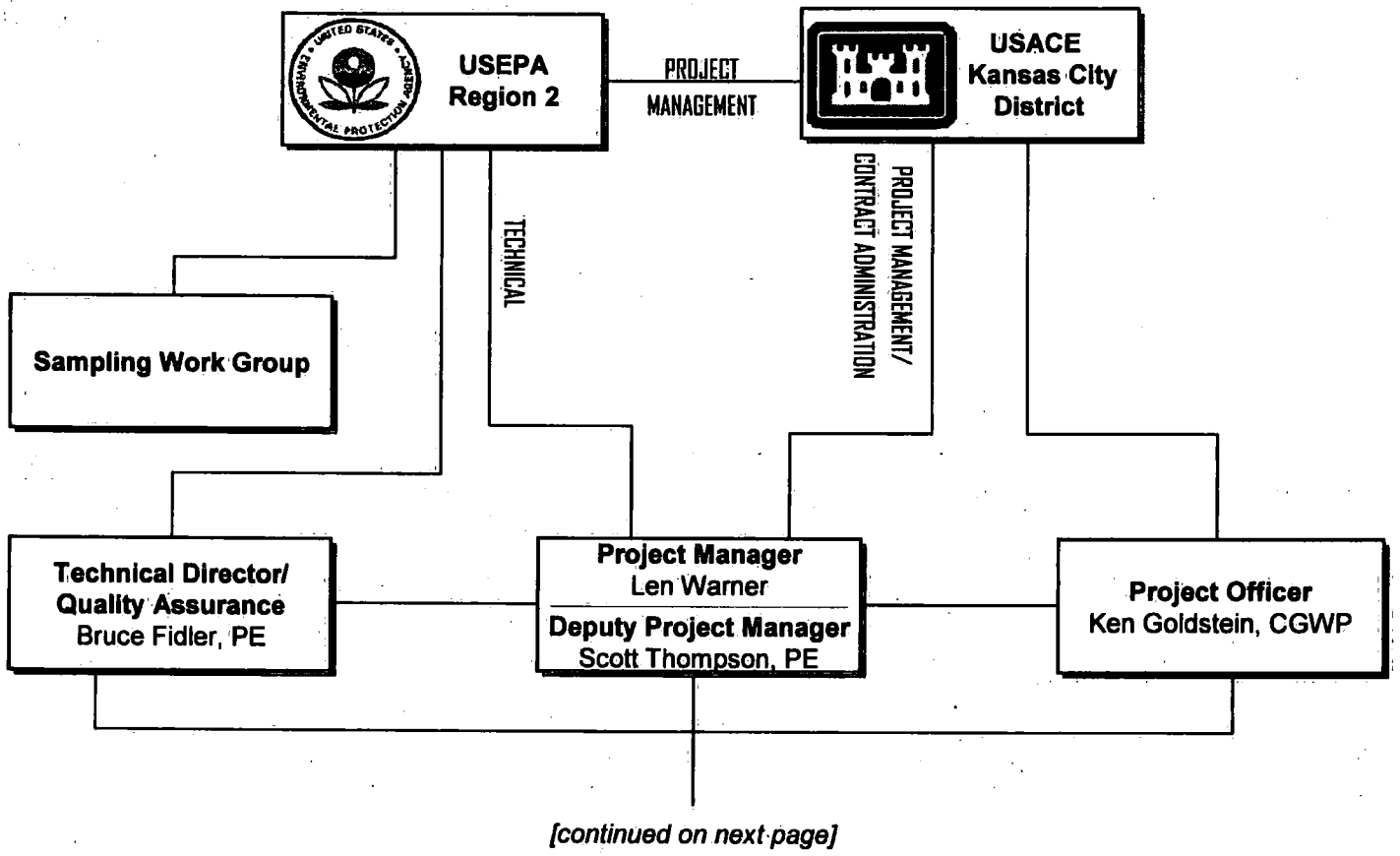
1.2 PROJECT/TASK ORGANIZATION

1.2.1 Overview

The project management team (see Figure 1) will consist of representatives from USEPA Region 2, USACE-KC, USACE-NY, NJDOT-OMR, Malcolm Pirnie, Inc. (Malcolm Pirnie), HydroQual, Inc., Battelle, and TAMS, *an Earth Tech company*. The USEPA Region 2 is the lead agency for Field Sampling Plan Volume 1 (Malcolm Pirnie, Inc., 2005a) and will provide project management. The USACE-KC will provide contract management and technical guidance. Malcolm Pirnie will be the primary contractor and will be responsible for developing and implementing the investigation and will provide project management to the other subcontractors. A Sampling Work Group, composed of six partner agencies and interested stakeholders, has been formed to advise the project management team on sampling issues.

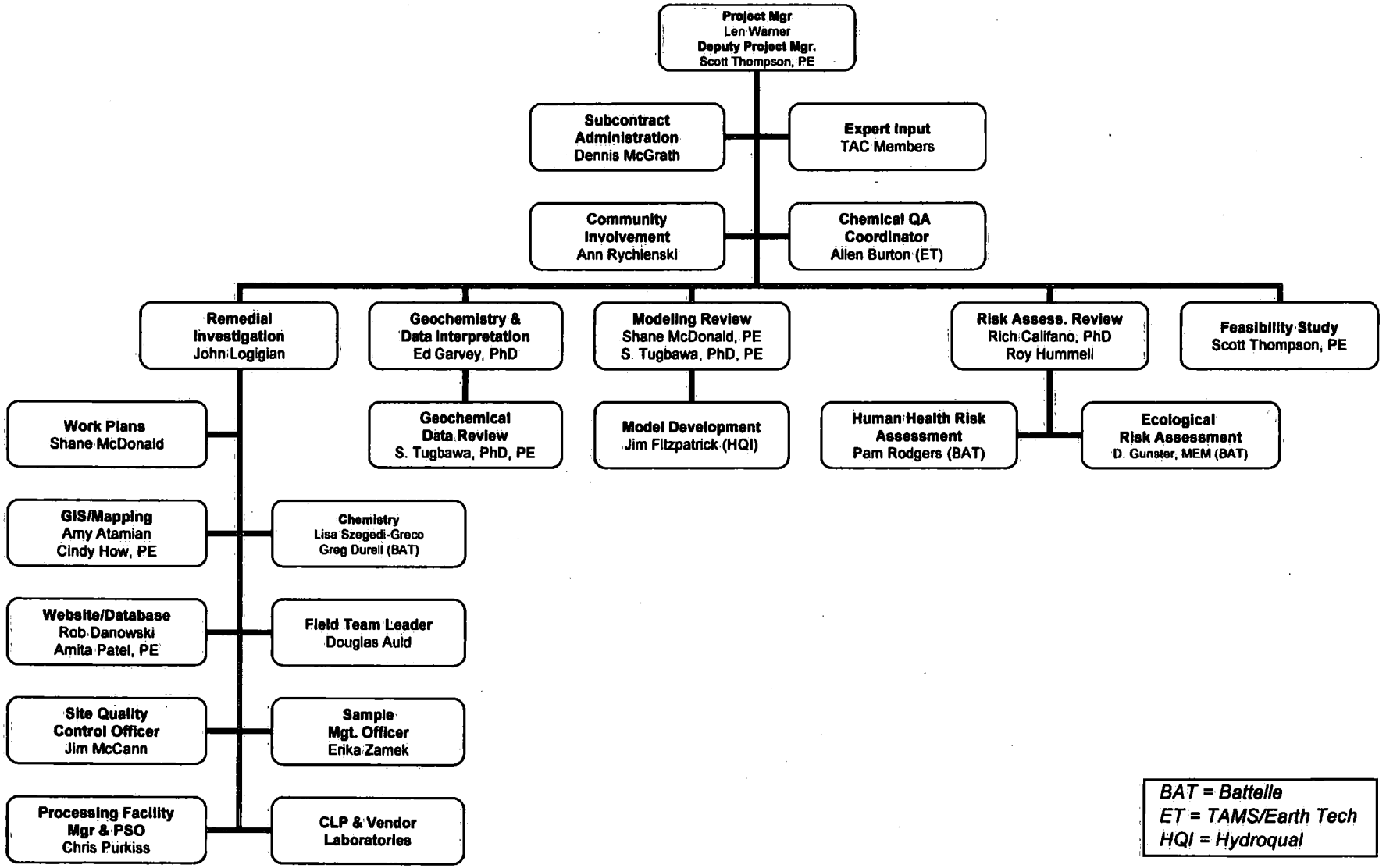
1.2.2 Project Management Structure

This section contains a description of the project organizational structure. Alice Yeh is the USEPA Project Manager with responsibility for the Passaic River project. Beth Buckrucker is the USACE-KC Project Manager, Lisa Baron is the Project Manager representing the NJDOT-OMR, and Scott Nicholson is the Project Manager representing the USACE-NY.



Lines of Communication between USEPA, USACE, and Malcolm Pirnie

MALCOLM PIRNIE	**Figure 1:** Lower Passaic River Restoration Site Project Team Organization Chart	QAPP Version: 2005/08/26



Lines of Communication between Malcolm Pirnie, Battelle, HydroQual, TAMS/EarthTech, Technical Advisory Committee

MALCOLM PIRNIE

Figure 1: Lower Passaic River Restoration Site Project Team Organization Chart (cont'd)

Malcolm Pirnie project team members are located primarily in the firm's Fair Lawn, NJ and White Plains, NY offices. Malcolm Pirnie project team members are also drawn from other regional offices, as appropriate. Additional project team members from other companies are subcontracted to Malcolm Pirnie; information is provided regarding subcontractor team members in Section 1.2.5 – Subcontractor Team Members. Contact information for key project personnel is posted on PREmis. The responsibilities of key project staff are summarized below:

- Bruce Fidler, PE, Technical Director (TD), is responsible for providing technical direction and overall strategy, facilitating consistency with other sediment investigations in the region, such as Newark Bay and Berry's Creek, and quality assurance of deliverables. The TD will:
  - Provide advice and input on the scope and sequencing of work.
  - Provide technical input for the preparation of deliverables such as work plans, reports, and technical memoranda, as well as other tasks performed under this contract.
  - Coordinate with members of the Quality Control Team (refer to Section 1.2.3).
  - Participate in QC reviews of submittals and prepare QC checklists.
  - Coordinate team attendance at independent peer reviews of project scientific deliverables, and preparation of responses..
  - Sort out technical advisory committee reviews and activities (refer to Section 1.2.3).
- Len Warner, Project Manager (PM), is primarily responsible for the development and implementation of the field investigation, including coordinating the community involvement, modeling, risk assessment (RA), and feasibility study (FS) work. As part of this responsibility, he will:
  - Lead the activities of the project team and the subcontractors.
  - Maintain budget and schedule surveillance and ensure timely submission of deliverables.
  - Communicate directly with the USEPA, the USACE, and stakeholders.
  - Approve reports and material for release to the USACE and other external agencies.
  - Oversee subcontractor performance.
  - Allocate resources and staffing to implement the project work.

- Scott Thompson, PE, Deputy Project Manager (DPM), reports directly to, and works with, the Malcolm Pirnie PM. As delegated, the DPM is responsible for interacting with the USEPA and USACE PMs, project team members, subcontractors, and stakeholders to ensure that the project is completed according to plan and in a timely manner. The DPM is accountable directly to the PM and is responsible for the logistics of project activities such as:
  - Preparing reports/products.
  - Coordinating office and field activities.
  - Timely submission of deliverables.
  - Scheduling activities.

- John Logigian, Field Investigation Leader, will be the Malcolm Pirnie contact person for all activities related to conducting the remedial investigation (RI). As such, he will be responsible for:
  - Directing the activities of personnel responsible for developing the planning documents, the website, the database, the Geographic Information System (GIS), and the field application.
  - Preparing reports/products.
  - Scheduling activities.
  - Coordinating with the USEPA and the USACE, as appropriate.

- Richard Califano, RA Leader, will be the Malcolm Pirnie contact person for activities related to conducting the risk assessment (RA). As such, he will be responsible for:
  - Providing technical support to Battelle for the RA.
  - Coordinating with the USEPA and the USACE, as appropriate.

- Shane McDonald and Solomon Gbondo-Tugbawa will be the primary Malcolm Pirnie contact persons for activities related to producing the Passaic River/Newark Bay numerical model. As such, they will be responsible for providing technical review of the HydroQual modeling activities.

- Scott Thompson, FS Leader, will be the Malcolm Pirnie contact person for activities related to conducting the FS. As such, he will be responsible for:
  - Evaluating data being collected.
  - Brainstorming the remediation options.
  - Providing feedback to the program based upon his findings and the data needs of the remediation options being considered.

1.2.3 Quality Control Team Structure

QC for the project will be provided by several personnel including the Site Quality Control Officer (SQO), quality reviewers, the project chemical quality assurance coordinator (QAC), and the technical advisory committee (TAC). Members of this Quality Control Team (QCT) are independent of the project team personnel. The roles and responsibilities of each QCT member are described below.

- Quality Reviewers will be identified by the TD and PM from among Malcolm Pirnie (or team subcontractor) senior technical staff, as appropriate to the particular technical deliverable. They will provide technical guidance and quality review to the project team and will review project plans and deliverables.
- Jim McCann, SQO is responsible for on-going supervision of project activities to ensure conformance to the planning documents and to evaluate the effectiveness of their requirements. The SQO will have access to any personnel (internal or subcontractors), as necessary, to resolve technical problems, and has the authority to recommend that work be stopped when that work appears to jeopardize the quality of the project efforts. The SQO will conduct regularly scheduled Technical System Audits (TSAs) of each type of field activity and will also be available to respond to any QA/QC problem. The SQO will be responsible for making sure that corrective actions called for as a result of a TSA are addressed. In addition, the SQO will be responsible for:
  - Monitoring the correction of quality problems and alerting task managers where similar problems might occur.
  - Developing and maintaining project QA files for the retention of sampling, monitoring, and field QA records.
  - Participating in QA audits and conducting TSAs.
  - Recommending changes to the PM to improve the effectiveness of the project in attaining its QA objectives for field, sampling, and monitoring activities.
  - Making sure that the planning documents are being followed.
  - Reviewing proposed additions and changes to this QAPP.
  - Reviewing deliverables for technical content and quality objectives.
  - Overseeing the QA of the Contract Laboratory Program (CLP) via the Regional Sample Control Center (RSCC) and subcontract laboratories, as well as data validators.
- Allen Burton, QAC, is responsible for monitoring the chemistry-related work being conducted for all programs [Comprehensive Environmental Response, Compensation,

and Liability Act (CERCLA) and Water Resources Development Act (WRDA)] involved in this project. As such, he will be responsible for:

- Reviewing project plans so that data collected for the various programs is comparable, useful to the majority of the entities involved, and collected in a format that is compatible with PREmis.
- Reviewing the Field Sampling Plan (FSP) Volume 1 (Malcolm Pirnie, Inc., 2005a) and Volume 3 (2005b) for applicability of the field sample collection methods (*e.g.*, filtered vs. non-filtered, time-weighted composites vs. grabs), determining which field data will be recorded in the field laptop (*e.g.*, sediment type, portion of the tidal cycle), sample frequency, depth, spatial distribution, and applicability of the analytical methods.
- Reviewing the QAPP for applicability of analytical methods and holding times; QC and response check requirements for the field and laboratory instruments; detection limits, action limits, and reporting limits; validation requirements; sample database storage requirements; electronic data deliverable (EDD) requirements; and data quality objectives (DQOs).
- Reviewing results of field audits, analytical laboratory performance and data validation, and recommending contingency or corrective measures, if necessary, to maintain data useability and defensibility.

- TAC Members: Refer to Table 1-1 for a listing of the current TAC members. It should be noted that as the project progresses, additional experts may be added. The purpose of the TAC is to support the technical credibility of the work conducted by the project team by providing technical guidance as well as independent review of the technical scope and direction of the project. In addition, TAC members will be responsible for:
  - Advising on recent technologies and methods.
  - Providing expert input to and review of various project plans.
  - Conducting expert review of observations, conclusions, and technical deliverables.
  - Assisting the team in preparing for and responding to independent peer reviews.

1.2.4 Field Team Members

- Mark McGowan, Certified Industrial Hygienist (CIH), Certified Safety Professional (CSP), Corporate Health and Safety Manager, serves as the administrator of the Corporate Health and Safety program. He is accountable directly to Malcolm Pirnie's President for project health and safety concerns and is responsible for:
  - Proper training for Malcolm Pirnie field personnel.

- Overseeing the Malcolm Pirnie medical monitoring program.
- Providing guidance on interpretation of exposure monitoring data.
- Determining the level of protective equipment to be used in the field.
- Evaluating compliance with the Health and Safety Plan (HASP) Core Document and task-specific addenda through regular audits of field activities.
- Approving the HASP and any task-specific addenda.

- Chris Purkiss, Project Safety Officer (PSO), reports directly to Malcolm Pirnie's Corporate Health and Safety Manager. The PSO will have access to any personnel or subcontractors, as necessary, to resolve health and safety problems, and will have the authority to stop work when that work appears to jeopardize safety. The PSO is responsible for identifying and prescribing appropriate protective measures. The PSO is responsible for:
  - Preparing the site-specific HASP Core Document and task-specific addenda.
  - Performing periodic health and safety audits.
  - Checking that health and safety procedures are observed in the field.
  - Monitoring personnel exposure to chemical toxins.
  - Developing emergency response procedures.
  - Monitoring for physical stress (*e.g.*, temperature).
  - Establishing personnel and equipment decontamination procedures.
  - Assigning alternate PSOs or designees in cases where more than one field team is operating at a time.

- Douglas Auld, Field Team Leader, is responsible for implementation of tasks performed as part of a given field event. If warranted, multiple field team leaders may be identified if multiple field work activities are scheduled concurrently. The Field Team Leader is responsible for:
  - Coordinating the work of Malcolm Pirnie and subcontractor field team members.
  - Mobilizing the necessary equipment and personnel to conduct the work.
  - Making sure that the planning documents are properly followed, including the standard operating procedures (SOPs).

- Members to be determined, a Field Activity Team, will be assembled from a qualified pool of personnel for each field event. The Field Activity Team is led by the Field Team Leader. The team is responsible for:

- Performing their assigned field sampling activities (as directed by the Field Team Leader).
- Make sure that the planning documents are properly followed, including the SOPs.

- Erika Zamek, Sample Management Officer (SMO), is tasked with the care and custody of environmental samples collected for the project. The SMO is responsible for:
  - Maintaining custody of the samples and preparing proper documentation of their transport to the laboratories.
  - Checking that the sample bottles are correctly labeled and the chain-of-custody (COC) forms and sample tags are properly filled out.
  - Maintaining project SMO files including COCs and bills of lading.
  - Making sure that the samples are properly preserved and custody sealed.
  - Checking that the samples are properly bagged and packed to minimize the potential for cross-contamination.
  - Coordinating sample delivery and receipt with the laboratory(ies).
  - Coordinating with the CLP and subcontractor laboratories to arrange for shipment of the samples.

1.2.5 Subcontractor Team Members

Several subcontractors will be utilized for performance of specific work activities associated with the Lower Passaic River Restoration Project. For a description of how subcontractors will be selected, refer to Section 6.0 (Subcontractor Management and Control) of the Final Quality Control Plan (QCP) (Malcolm Pirnie, Inc., 2003b). The following is a list of services to be subcontracted for the site:

- Modeling: HydroQual is responsible for developing models including the Hydrodynamic Model, the Sediment Transport Model, the Chemical Fate and Transport Model, and the Bioaccumulation Model. HydroQual will also provide a technician to the field team to help coordinate sample collection, establish a connection between the modelers and the field data collection, and provide additional technical support as needed.
- Risk Assessment: Battelle is responsible for conducting the ecological and human health RA. Battelle is also responsible for providing additional technical support as needed.

- Laboratory Analysis: The subcontract laboratories will include Axys Analytical, Severn Trent Laboratories (STL), and Outreach Analytical. Other laboratories may be added as necessary. These laboratories will be responsible for the analysis of samples for non-CLP parameters and/or media. Note that not all analyses will be conducted by subcontractor laboratories; some will be conducted by CLP laboratories or USEPA's DESA laboratory.
- Boat and Coring Services: The boat and coring services subcontractor is AquaSurveys, Inc. The on-water sediment coring services subcontractor(s) will be responsible for mobilizing all required equipment and personnel to the site, positioning over coring locations, core collection, handling, preservation, and delivery to the field office(s). The location and riverbed elevation of all core samples will be determined by the subcontractor using global positioning system (GPS) equipment.
- Data Validation Services: To be determined. The subcontractors will be responsible for validating non-CLP data as well as any CLP data that exceeds RSCC's capacity. They will also be responsible for writing validation reports and data usability reports, as well as making data changes and marking data qualifiers on the EDD module on PREmis.

1.3 PROBLEM DEFINITION/BACKGROUND

The Passaic River surface water and sediments are contaminated with a variety of chemicals including dioxins/furans, polychlorinated biphenyls (PCBs), organic pesticides, polycyclic aromatic hydrocarbons (PAHs), and inorganic chemicals such as mercury and lead. The contaminated sediments underlying the Passaic River are of concern to various federal and state regulatory agencies because they can have:

- Ecological health effects.
- Human health effects.
- Economic impacts on navigational dredging disposal costs.
- Economic impacts from lost use (*e.g.*, due to consumption advisories).

The problem definition, site background, and historic information are fully described in the Work Plan (WP) (Malcolm Pirnie, Inc., 2005c). Sections 1 (Introduction), 2 (Environmental Setting), 3 (Work Plan Rationale), and 4 (Preliminary Evaluation) of the WP (Malcolm Pirnie, Inc., 2005c) summarize the history of the Study Area, evaluation of historical sediment data, and the preliminary Conceptual Site Models (CSMs). The CSMs identify the sources and mechanisms of potential contamination

release within the Study Area and the possible pathways whereby human and ecological receptors may be exposed to sediment contamination. The CSMs will be updated based on ongoing data collection and analysis of historical geochemical data. Figure 2 provides a map of the site location. The river mile (RM) 0.0 established for the LPRRP uses two lighthouses, one located in Essex County, NJ (lat = 40.707725; long = 74.118945) and the other located at Kearny Point in Kearny, NJ (lat = 40.712119; long = 74.115551), as markers. An imaginary line drawn between these lighthouses is assigned as RM 0.0 for the LPRRP.

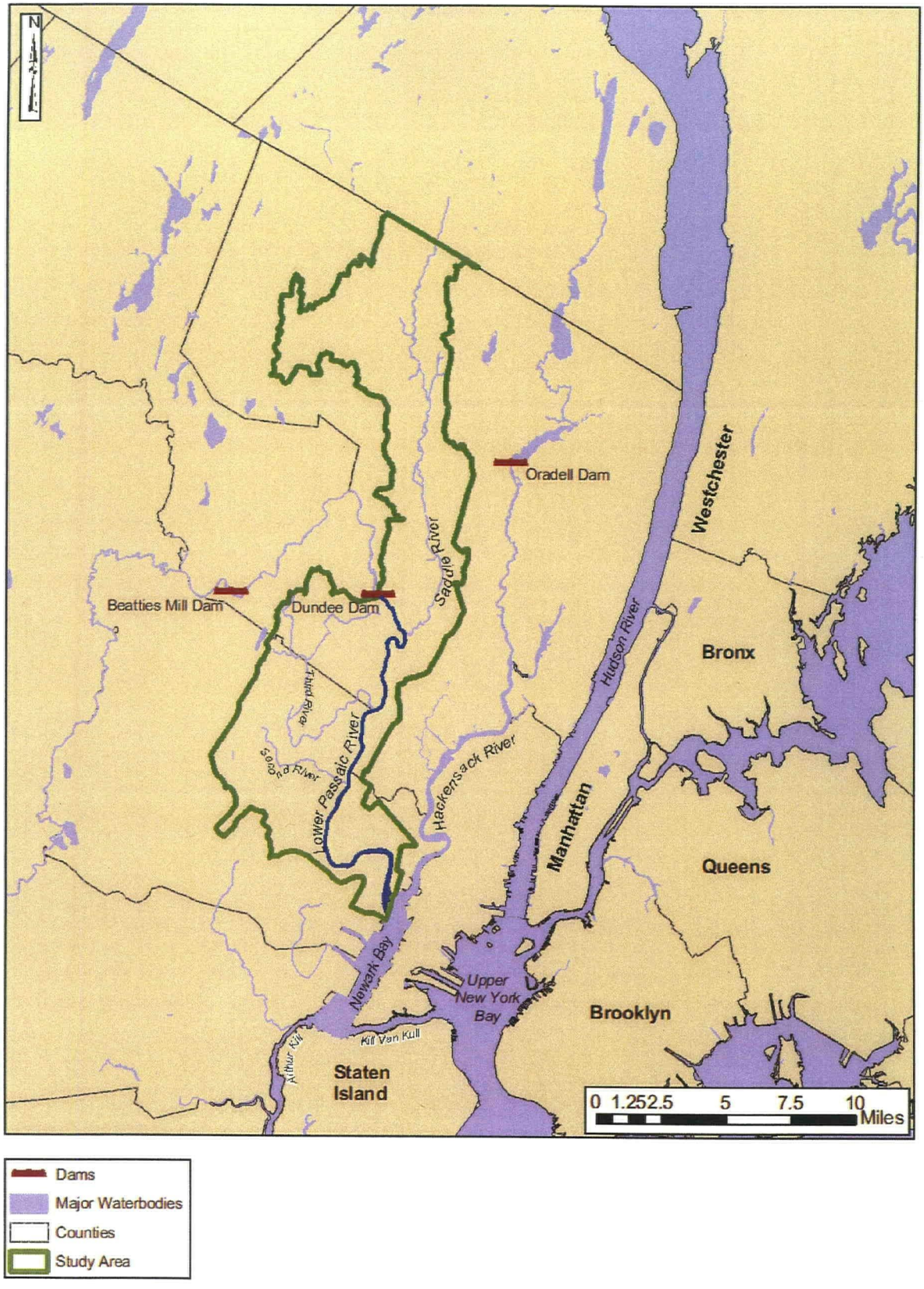


Figure 2: Lower Passaic River Study Restoration Project – Site Location Map

1.4 PROJECT/TASK DESCRIPTION

1.4.1 Task Description

The project will include the sampling and analysis of sediment, water, and biota for wet chemistry, geotechnical parameters, and physical properties as well as chemicals of potential concern (COPCs) and chemicals of potential ecological concern (COPECs). The sampling and analysis of sediment, surface water, and/or biota described in this QAPP and subsequent amendments will center primarily on the lower 17 miles of the Passaic River and its tributaries, but will also extend, as appropriate, into connected water bodies such as the Hackensack River, Newark Bay, Arthur Kill, and the Kill van Kull.

A full description of the project tasks are given in the WP (Malcolm Pirnie, Inc., 2005c). Planned sampling activities are fully described in FSP Volume 1 (Malcolm Pirnie, Inc., 2005a), FSP Volume 2 (to be finalized in 2006), and FSP Volume 3 (Malcolm Pirnie, Inc., 2005c).

1.4.2 Work Schedule

Water and sediment samples will be collected during the summer and fall of 2005. The sampling program will continue throughout 2006 and will expand to include the collection of biota samples. A detailed project schedule is posted on PREmis under the "Project Management" header, and the "Schedule" sub-header. The project schedule is updated regularly (*e.g.*, monthly) based on discussions with the project team members (*i.e.*, USACE, USEPA, NJDOT-OMR, and subcontractors) as well as the effect of seasonal and weather considerations on field sampling activities. The analytical laboratory requirements for the 2006 sampling events (including biota programs), will be revisited following the review of the data collected during the 2005 sampling events and the approval of FSP Volume 2.

1.5 QUALITY OBJECTIVES AND CRITERIA

This section discusses the performance, measurement, and acceptance criteria for the data to be collected for this project. As such, it includes the following sections:

- Project Data Quality Objectives.

- Precision, Accuracy, Representativeness, Completeness, and Comparability.
- Desired Method Sensitivity, including project action levels (ALs) and reporting limits (RLs) for the parameters of interest.

1.5.1 Project Data Quality Objectives

The overall QA objective is to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide scientifically sound results that can be used to make defensible decisions (*i.e.*, data of known and documented quality that are adequate for their intended use). In this section, the QA objectives that are required for the data collected during the Lower Passaic River Restoration Project are developed and specifically identified. The DQO process, which is a systematic planning process, takes into consideration the intended data use, the available laboratory and field analysis procedures, and the available resources. The process' end result is the development of quality requirements for each data collection activity. The DQOs for the project are documented in Attachment 1.1. Based upon these DQOs, analytical methods that are capable of supporting the DQOs were selected (Refer to Section 2.4 – Analytical Methods). The QA objectives for the analytical methods were also determined (Refer to Section 2.5 – Quality Control).

The historical data evaluations, geochemical evaluations, and field sampling programs described in the WP (Malcolm Pirnie, Inc., 2005c) and FSP Volume 1 (Malcolm Pirnie, Inc., 2005a), Volume 2 (to be finalized in 2006), and Volume 3 (Malcolm Pirnie, Inc., 2005b) are designed to address the problem statement and Fundamental Questions presented in DQO Steps 1 and 2. The problem statement in DQO Step 1 centers on the following objectives of the Lower Passaic River Restoration Project investigation activities ("the Study"):

- To characterize the nature and extent of contamination in the Lower Passaic River.
- To characterize the mechanisms governing long-term fate and transport of site contaminants.
- To assess the human health and ecological risks posed by the contamination in the Lower Passaic River.
- To characterize the function and structure of candidate restoration sites in the Lower Passaic River watershed.

- To evaluate remedial alternatives that meet both CERCLA and WRDA selection criteria to address unacceptable human health/ecological risks and provide for restoration within the Lower Passaic River watershed; as well as to evaluate options for reducing costs associated with dredging contaminated harbor sediments originating from the Passaic River.
- To support development of a natural resource damage assessment (NRDA) under CERCLA.

The following Fundamental Questions need to be answered during the investigation to meet these objectives:

1. If we take no action on the River, when will the COPCs and COPECs recover to acceptable concentrations?
2. What actions can we take on the River to significantly shorten the time required to achieve acceptable or interim risk-based concentrations for human and ecological receptors?
3. Are there contaminated sediments now buried that are likely to become exposed following a major flood, possibly resulting in an increase in contaminants within the fish/crab populations?
4. What actions can we take on the River to significantly improve the functionality of the Lower Passaic River watershed?
5. If the risk assessments for Newark Bay demonstrate unacceptable risks due to contaminant export from the Passaic River, will the plan proposed to achieve acceptable risks for Passaic River receptors significantly shorten the time required to achieve acceptable or interim risk-based concentrations for receptors in Newark Bay, or will additional actions be required on the Passaic River?[1]
6. What actions can we take on the River to significantly reduce the cost of dredged material management for the navigational dredging program?
7. What actions can we take to restore injured resources and compensate the public for their lost use?

1.5.2 Precision, Accuracy, Representativeness, Completeness, and Comparability

To measure and control the quality of analyses, certain QA parameters are defined and utilized in data analysis activities. These parameters are defined below. The QA/QC required for the parameters to be analyzed under the USEPA CLP is contained in the sections of the USEPA CLP Statement of Work (SOW). The required QA/QC for the

1 This question is shared with the RI/FS for the Newark Bay Study, since the actual benefits of such reduction will need to be jointly determined. A similar question to address the adequacy of any future Newark Bay plan toward achieving Passaic River goals may be included in the Newark Bay RI/FS.

non-CLP laboratory test methods including the frequency, reporting limits, and required actions to be taken if QC criteria are not met are given in Attachment 3. Detailed information on the CLP methods and QA/QC criteria can be found in the USEPA CLP SOW found on the USEPA CLP website at http://www.epa.gov/superfund/programs/clp/.

Precision

Precision measures the reproducibility of data or measurements under specific conditions. Precision is a quantitative measure of the variability of a group of data compared to their average value. Duplicate precision is stated in terms of relative percent difference (RPD) or absolute difference between two measurements. Measurement of precision is dependent upon sampling technique and analytical method. Field duplicate and laboratory duplicate samples will be used to measure precision for project samples. Both sampling and analysis will be as consistent as possible. For a pair of measurements, RPD (or absolute difference) will be used, as presented below:

$$RPD(\%) = \frac{|D_1 - D_2|}{\left[\frac{(D_1 + D_2)}{2}\right]} \times 100$$

where: D_1 and D_2 = the two replicate values.

RPD will meet EPA CLP requirements, when applicable, or the QA requirements listed in Attachment 3.

Accuracy

Accuracy measures the bias in a measurement system. Sources of error include the sampling process, field contamination, preservation, handling, shipping, sample matrix, sample preparation, and analysis technique. To evaluate whether the sampling process has introduced a bias into the sample results, rinsate blanks and trip/field blanks will be collected and analyzed, where appropriate. Analytical accuracy will be assessed through surrogate spike, matrix spike, laboratory control and/or quality check samples. In general, accuracy is measured in terms of percent recovery (%R):

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

where: SSR = spike sample result
SR = sample result
SA = spike added from spiking matrix

Refer to Attachment 3 and the CLP SOW for the laboratory analytical method accuracy requirements.

Representativeness

Representativeness expresses the degree to which data accurately and precisely reflect a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design and implementation of the sampling program and proper laboratory protocol. The sampling network created for this project was designed to provide data representative of site conditions. During the development of the sampling network, consideration was given to the past history of contamination in the Study Area, existing analytical data, physical setting, and processes. The rationale used in developing the sampling network is discussed in detail in the FSP. Representativeness will be satisfied by determining that the FSP is followed, proper sampling techniques are used, proper analytical procedures are followed, and holding times for the samples are not exceeded in the laboratory.

It is not intended that the data set collected during the summer and fall of 2005 will fully represent the entire study area. These data are intended to begin characterization of the system and to allow the modelers to begin the modeling process. It is intended that the use and representativeness of these data will be evaluated during data gap analyses conducted during the winter of 2005-2006. Based on the data gap analyses, the 2006 sampling program will be designed.

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the laboratories used for this project will provide data that meet the QC acceptance criteria for 90 percent, or more, of all samples analyzed. Following the completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness (\%)} = \frac{\text{number of validated data}}{\text{number of samples collected for each parameter analyzed}} \times 100$$

As described in later sections of this QAPP, the data validation process will be used to determine the quality of analytical data generated.

The completeness acceptance criterion for samples collected in the field will be 95 percent of the quantity of samples planned for collection in the FSP. The final FSP will contain a detailed description of the planned number of samples for 2005. Corrective action will be implemented to re-collect samples where necessary and possible (*e.g.*, modifying a planned sample location, sample jars broken during shipment). Electronic sample receipt checklists will be used to determine, as soon as possible, whether any problems during sample shipment would necessitate recollection of samples.

Comparability

Comparability expresses the confidence with which one data set can be compared to another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data are expected to provide comparable data. It should be noted that the majority of the historical data was collected approximately 10 years ago. Due to advances in analytical instrumentation and methodology, it is likely that analyses being performed as part of this project will utilize methodologies that were not available at the time the historical samples were analyzed. As the new data are received, it will be

determined how comparability will be evaluated between the historic and current data sets. To the extent practicable, the comparability of these data sets will be evaluated on a parameter by parameter basis, considering the biases in different test methods as well as the different advancements that have been made for different parameters. For example, metals analyses have changed very little over the past decade while dioxin analytical methods have changed significantly with the use of additional surrogates and the advent of high resolution mass spectrometry.

1.5.3 Desired Method Sensitivity

This section discusses measurement performance criteria and desired method sensitivity. Depending on the use of the data, specific RLs will be required for each parameter. To establish RL requirements, certain terms must first be defined.

- Method Detection Limit (MDL): The MDL is the concentration of a particular compound that can be detected by a particular method. The concentration must be greater than zero and the compound must be detected with at least a 99% confidence level. The laboratory MDL must be low enough to support the RL for the test parameter.
- RL: The RL is the lowest concentration typically reported for a specific compound in a sample after corrections have been made for dilution factors, weight (for solid samples), and percent moisture. Note that in some instances laboratories are able to report values below the RL; these concentrations are qualified to denote further describe data usability below the RL. It should be noted that RLs are highly dependent on matrix effects. A calibration point at the RL is typically included in a laboratory's initial calibration.

Attachment 2 contains a compilation of representative human health and ecological risk-based ALs for the COPCs/COPECs identified in the Pathways Analysis Report (PAR) prepared for the LPRRP (Battelle, 2005). The ALs were compiled and evaluated as the basis for the required RLs listed in Tables 2-1 through 2-21.

For the water and sediment sampling scheduled for 2005, some of the inorganic and organic chemical test data will be obtained through the USEPA CLP or through USEPA's DESA laboratory. The USEPA CLP has extensive quality assurance requirements to document data quality and assist laboratories to produce data that are technically sound. The required sample quantitation limits for these data will be based upon the USEPA CLP capabilities. Under the CLP flexibility clause, lower quantitation

limits will be requested to address the risk assessment requirements of the project. After sampling planned for 2005 is completed, the data collected will be evaluated and it will be determined if it is necessary to investigate more specialized methods with potentially lower quantitation limits for subsequent data acquisition activities.

Laboratory RLs for tissue have not been included at this time, since biota samples will not be collected during the phase of sampling planned for 2005. RLs for biota will be included in a future revision/amendment of the QAPP, in coordination with FSP Volume 2.

Tables 2-1 through 2-4 list the laboratory target RLs for the chemical analyses of sediment and water samples that will be conducted through USEPA CLP. The organics include PCBs (Aroclors), Target Compound List (TCL) volatile organic compounds (VOCs), and TCL semivolatile organic compounds (SVOCs), including PAHs. The inorganics include Target Analyte List (TAL) metals and cyanide. Table 2-5 lists target RLs for PCB congeners based upon method 1668A. Current plans are to obtain all the PCB congener analysis through a single non-CLP laboratory (Axys Analytical) to maximize data consistency. The required laboratory RLs and the quality requirements for the non-CLP laboratory tests, listed in Tables 2-5 through 2-21, are provided in the tables in Attachment 3. The RLs presented in the QAPP were selected to address the risk assessment, modeling, and engineering requirements of the project in a technically sound and reasonable manner. The target RLs (given in Tables 2-1 through 2-21 and Attachment 3) were generally selected to be at or below the lowest risk assessment AL for the COPCs/COPECs, as shown in Attachment 2. For some parameters, such as dioxin/furans, PCBs, and several PAHs and pesticides, it was necessary to base the reporting limits on the quantitation limits achievable by the available laboratory methods, rather than ALs. As described in FSP Volume 1 for the water column sampling program, large volume samples will be collected for dioxin/furans, PCB congeners, and pesticides, which will achieve lower reporting limits for these critical COPCs.

1.6 SPECIAL TRAINING AND CERTIFICATIONS

Any specialized training requirements necessary to complete the project will be documented to ensure that the specific skills have been obtained, verified, and updated as necessary.

1.6.1 Training

Required training will be documented for all personnel, including subcontractors, performing functions requiring training. The Equipment Manager will have training as described in Section 2.6.1 – Preventative Maintenance and Instrument Calibration – Field Instruments. Project-specific health and safety training, such as training mandated by Occupational Safety and Health Administration (OSHA) regulations, training for shipping hazardous materials mandated by the Department of Transportation (DOT), and training for navigating vessels mandated by the United States Coast Guard (USCG), and/or others will be obtained as specified within the Project HASP (Malcolm Pirnie, Inc., 2005e).

1.6.2 Certification

Training and certification will be obtained, wherever necessary, for personnel prior to their involvement in the field sampling activities. No person will be allowed to perform tasks that require specific training without the respective current certification on file. These certifications will be documented and scanned into the project database (PREmis).

1.7 DOCUMENTS AND RECORDS

Requirements for the storage of documents and records can be found in the QCP. PREmis, an internal project database, was developed to collect, store, manage, and report all information gathered during the Lower Passaic River Restoration Project. PREmis is a centralized, web-based portal to the various forms of electronic information collected and stored for this project. Refer to Section 2.10 – Data Management for a more detailed description of PREmis. Public information is uploaded from PREmis to the public website, www.ourPassaic.org.

The subcontract laboratories must keep records of both raw and processed data generated on samples submitted on file. The laboratories' data record keeping procedures must be documented in the laboratory quality manual.

Further details concerning the project Documents and Records requirements are also discussed in Section 2.10 on Data Management.

2.0 DATA GENERATION AND ACQUISITION

This group of quality elements addresses measurement system design and implementation, including appropriate methods for sampling, analysis, data handling, and QC documentation.

2.1 SAMPLING PROCESS DESIGN (EXPERIMENTAL DESIGN)

Environmental sampling includes the collection of surface water, sediment, biota, soil, porewater, and groundwater samples; several geophysical, water quality, and sediment transport surveys will also be performed. Project sampling and field documentation procedures, as well as the objectives of each sample task, are provided in detail in the Lower Passaic River Restoration Project WP (Malcolm Pirnie, Inc., 2005c) and FSP Volume 1 (Malcolm Pirnie, Inc., 2005a), Volume 2 (to be finalized in 2006), and Volume 3 (Malcolm Pirnie, Inc., 2005b). This QAPP will be revised once FSP Volume 2 is issued. The purpose of the FSP is to ensure that samples are collected, handled, and documented correctly prior to analysis. See Section 5 (Field Investigation Tasks) of the WP (Malcolm Pirnie, Inc., 2005c), Section 3 (Field Tasks) of FSP Volume 1 (Malcolm Pirnie, Inc., 2005a), and Section 4 (Field Tasks) of FSP Volume 3 (Malcolm Pirnie, Inc., 2005b) for a listing of sampling activities, media to be sampled, types of analyses to be performed, and the number and location of samples to be collected. Attachment 1.2 summarizes the proposed sampling design described in FSP Volume 1 (Malcolm Pirnie, Inc., 2005a). It includes a list of project data needs, the associated data user (*e.g.*, geochemist, modeler, engineer, and/or risk assessor), the sampling program designed to meet each data need, media to be sampled, and the test parameters desired.

2.2 SAMPLING METHODS

The sampling procedures for sediment cores and surface water samples are provided in detail in the Lower Passaic River Restoration Project WP (Malcolm Pirnie,

Inc., 2005c) and FSP Volume 1 (Malcolm Pirnie, Inc., 2005a). Section 2.3 – Sample Handling and Custody, further discusses sampling requirements.

2.3 SAMPLE HANDLING AND CUSTODY

Sample custody procedures ensure the timely, correct, and complete analysis of each sample for all parameters requested. A sample or evidence file is considered to be in someone's custody if it:

- Is in his/her possession;
- Is in his/her view, after being in his/her possession;
- Is in his/her possession and has been placed in a secured location; or
- Is in a designated secure area.

Sample custody documentation provides a written record of sample collection and analysis. The sample custody procedures provide for specific identification of samples associated with an exact location, the recording of pertinent information associated with the sample, including time of sample collection and any preservation techniques, and a COC record which serves as physical evidence of sample custody. Custody procedures will be similar to the procedures outlined in the USACE's Requirements for the Preparation of Sampling and Analysis Plans (USACE, 2001) and the USEPA's Contract Laboratory Program Guidance for Field Samplers (USEPA, 2004a). The COC documentation system provides the means to individually identify, track, and monitor each sample from the time of collection through final data reporting. Sample custody procedures are developed in three areas: sample collection, laboratory analysis, and final evidence files, which are described below.

2.3.1 Field Sample Handling and Custody

Field records provide a means of recording information for each field activity performed at the site. Chain of custody procedures document pertinent sampling data and all transfers of custody until the samples reach the analytical laboratory. The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the chain of custody intact. Refer to SOP No. 1 in

Attachment 4 on Sample Management. SOP No. 2 in Attachment 5 covers sample preservation procedures. Tables 3-1 through 3-6 list the specific sample preservation requirements for each test method and sample matrix.

2.3.2 Field Procedures

a) The field sampler is personally responsible for the care and custody of the samples until they are transferred to the SMO or until they are properly dispatched. As few people as possible should handle the samples.

b) The Field Team Leader, or designee, is responsible for entering the proper information in the field laptop at each sample location, including all pertinent information such as sample identification number, method of sample collection, date and time of sample collection, type of analysis, and description of sample location. Refer to the FSP for more detail regarding the laptop field application. The information entered into the field laptop will be transmitted via wireless technology to the PREmis database; this information will be used to generate an electronic COC.

c) All sample bottles will be labeled with the project code, sample number, matrix, type of analysis required, and preservation requirements.

d) The samples will be properly preserved, bagged, and packed into coolers. SOP No. 2 in Attachment 5 contains the proper preservation techniques. The original COC form will be placed into the lead cooler, copies of the COC form will be placed in all other coolers, and the coolers will be shipped to the laboratory.

e) The SQO or his designee will review all field activities to determine whether proper custody procedures were followed during the field work and if additional samples are required.

2.3.2.1 Field Records

Refer to the FSP for the procedure on documenting field activities. The field laptop will provide the means of recording data collection activities. Entries will be described in as much detail as possible so that persons going to the site can reconstruct a particular situation without reliance on memory. At the beginning of each day, the date, start time, weather, names of all sampling team members present and level of personal protection being used will be entered. The names of visitors to the site and the purpose of their visit will also be recorded. All field measurements, as well as the instrument(s) used (including the instrument's assigned Passaic River project barcode, located on the back of all field equipment) will be noted.

Samples will be collected following the sampling procedures documented in the FSP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, associated rinsate blanks, and number of containers. Observations such as sampling conditions or any problems will also be recorded. Sample identification numbers will be assigned at the time the data are entered into the laptop. Field duplicate samples, which will receive a unique sample identification number, are "blind" to the laboratory and will be identified under the sample description so that they can be associated with their respective samples by project staff. Matrix Spike (MS)/Matrix Duplicate (MD), and MS/Matrix Spike Duplicate (MSD) samples will also be noted, but do not receive unique sample identification numbers.

2.3.2.2 Sample Identification

The documentation system for laboratory samples will be based on the sample documentation system described in USACE (2001) and USEPA (2004a) guidance documents. Sample identification procedures are also described in the FSP. All samples collected will have a label that contains the following information:

1. Project name and/or number.
2. Field ID or sample station number.
3. Designation of sample as grab or composite.
4. Sample matrix.
5. Sample preservation notes.
6. Analytical parameters.

2.3.3 Chain of Custody Procedure

At the time of sampling, an electronic COC form will be generated by PREmis based on the information entered into the field laptop. The COC form generated by PREmis will be in the same format as those generated by FORMS II Lite. In addition, PREmis will be programmed to transmit information to FORMS II Lite that the latter application needs, in order for the XML files to be transmitted to CLP's Sample Management Office on the pre-determined schedule (at the close of each case). The hard copies of the COCs generated by PREmis (in FORMS II Lite format) will be sent to the

laboratories and RSCC daily. These procedures are by arrangement with RSCC (Jennifer Feranda, personal communication, February 10, 2004). The following information will be recorded on the COC form (note that most of this information will be filled in by PREmis when the COC is generated; the signatures will be in ink).

1. Project name and/or project number.
2. Signature of SMO or designee.
3. Sampling station number.
4. Date and time of collection.
5. Grab or composite sample designation.
6. Sample matrix.
7. Sampling location description.
8. Field identification number.
9. Analyses required.
10. Preservation technique.
11. Signatures and dates for transfers of custody.
12. Air express/shipper's bill of lading identification numbers.

The COC form serves as an official communication to the laboratory detailing the particular analyses required for each sample. The COC record will accompany the samples from the time of sampling through all transfers of custody. It will be kept on file at the laboratory where samples are analyzed and archived. Three copies of the COC form are created; one copy is retained by the Field Team Leader and two are sent to the laboratory. The SMO or designee completes a COC record to accompany each shipment from the field to the laboratory. The completed COC is put in a zip-lock bag and taped to the inside cover of the sample shipping container. If there are more than one container in a shipment, copies of the COC forms will be placed in each container. The container is then sealed with custody seals and custody is transferred to the laboratory.

2.3.4 Transfer of Custody and Shipment

The custody of samples must be maintained from the time of sampling through shipment and relinquishment to the laboratory. Instructions for transferring custody are given below:

1. All samples are accompanied by a COC. When transferring custody of samples, the individuals relinquishing and receiving will sign, date, and note the time on the COC. This form documents sample custody transfer from the SMO or designee, through the shipper, to the analytical laboratory. Since a common carrier will usually not accept responsibility for handling COC forms, the name of the carrier is entered under "Received by", the bill-of-lading number is recorded in the comments section, and the COC form is placed in a zip-lock plastic bag and taped to the inside lid of the lead shipping cooler. Copies of the COC forms will be placed in each additional cooler in a shipment.
2. Samples will be packaged for shipment and either picked up at the site by the laboratory or dispatched to the appropriate laboratory via overnight delivery service. SOP No. 1 in Attachment 4 contains the proper sample packaging techniques. A separate COC record must accompany each shipment. Shipping containers will be sealed for shipment to the laboratory. Two custody seals will be applied to each cooler to document that the container was properly sealed and to determine if the container was tampered with during shipment. The custody seals will be placed on the coolers in such a manner that the custody seal would be broken if the cooler were opened (*i.e.*, diagonally opposite corners of the cooler lid).
3. The original COC (and a copy for CLP laboratories) will accompany the shipment. A copy will be retained by the Field Team Leader.
4. If the samples are sent by common carrier or air freight, proper documentation must be maintained. For example, the bill of lading must be retained by the Field Team Leader.

2.3.5 Laboratory Custody Procedures

The laboratory custody procedures will be equivalent to those described in the latest edition of the CLP SOW. The following will be addressed in the laboratory custody SOPs:

- A designated sample custodian accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory. An example Sample Receipt checklist is given in Attachment 6. The CLP laboratories will send a copy of the sample receipt checklist to USEPA's RSCC, while the subcontract laboratories will fill out the form electronically on PREmis.
- Once the samples have been accepted by the laboratory, checked and logged in, they must be maintained in accordance with laboratory custody and security requirements.
- To ensure traceability of samples while in the possession of the laboratory, a method for sample identification that has been documented in a laboratory SOP will be used to assign sample numbers.

- The following stages of analysis must be documented by the laboratory:
  - Sample Extraction/Preparation.
  - Sample Analysis.
  - Data Reduction.
  - Data Reporting.
- Laboratory personnel are responsible for the custody of samples until they are returned to the sample custodian.
- When sample analyses and QA checks have been completed in the laboratory, the used portion of the sample must be stored or disposed of in accordance with the protocols specified in the CLP SOW or the subcontract agreement. Identifying labels, data sheets, COCs, and laboratory records will be retained until analyses and QA checks are completed in accordance with the protocols specified in the CLP SOW or the subcontract agreement.

2.3.6 Final Evidence Files

This is the final phase of sample custody. The COC records and sample analysis request form copies are archived in their respective project files. Laboratory custody forms, sample preparation and analysis logbooks, and data packages will become part of the laboratory final evidence file. Other relevant documentation including records, reports, and correspondence, logs, pictures, and data review reports will be archived by Malcolm Pirnie.

2.3.7 Sample Holding Times

Information on sample holding times and required preservation for each test method and matrix are given in Tables 3-1 through 3-6.

2.4 ANALYTICAL METHODS

All samples collected during field sampling activities for the Lower Passaic River Restoration Project will be analyzed either through the USEPA CLP program or via subcontract laboratories. For non-CLP parameters, the analysis will be performed by laboratories qualified in the analytical methods and, where applicable, certified through the programs listed below. Each subcontract laboratory utilized for the project will undergo an evaluation to determine if their experience and capability in the requested analytical methods are appropriate for the project

- National Environmental Laboratory Accreditation Program (NELAP);

- NJDEP;
- NOAA National Status and Trends (NS&T);
- USEPA CLP – Qualified Laboratory; and/or
- USACE.

When possible the test methods selected were either USEPA methods or national consensus methods, such as those published by ASTM, or in Standard Methods for the Examination of Water and Wastewater.

The analytical methods were selected based on the DQOs established for the project. Depending on the use of the data, different analytical methods may be required for the same parameters in order to achieve different RLs. This is because the RL may change depending on the use of the data. For example, lower RLs will be required for samples used to delineate the vertical and horizontal extent of contamination than for samples collected within a known hot spot. The analyte groups and analytical methods to be used for the studies on samples of sediment and water planned for 2005 are given in Tables 4-1 through 4-5. The analytical methods for sediment and water samples planned for 2006 will likely be the same as those used in 2005. However, the applicability of the methods will be evaluated during the data gap analysis and prior to determining the 2006 sampling program. The analytical methods for tissue samples are not given since they will not be collected until 2006. The analytical methods appropriate for required tissue analysis will be included in a revision/addendum to the QAPP, developed in coordination with FSP Volume 2.

The following is a description of the techniques proposed for the key laboratory analytical methods. Depending on the capabilities of laboratories employed to support the project, modifications may be made to the specific test methods and quality assurances described herein so long as the data quality is sufficient to meet project objectives, and all modifications are documented and approved by the SQO.

2.4.1 Inorganic Methods

Methods for the inorganic analyses are listed in Table 4-1. Many of the metals analyses for surface water and sediment samples collected during the 2005 sampling event will be performed by the methods described in the Laboratory SOW for the USEPA

(2004b) CLP Multi-Media, Multi-Concentration, Inorganic Analytical Services for Superfund (ILM05.3 or latest version). TAL metals reported under this program include aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, thallium, vanadium, and zinc, plus cyanide. In addition, titanium is also being requested under the CLP flex clause, since it was identified as a COPC/COPEC.

Analytical techniques used are described in the CLP SOW and include Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) and ICP-Mass Spectrometry (ICP-MS). (The ICP-MS option will be utilized for metals for which it is necessary to achieve the RLs specified in Table 2-1). Total mercury will be determined by Cold Vapor Atomic Absorption (CVAA). Total cyanide will be measured in water and sediment by a colorimetric method described in the CLP SOW.

Selected water and sediment samples, as described in FSP Volume 1 (Malcolm Pirnie, Inc., 2005a), will also be analyzed for trace metals and metals species including: trace mercury, methyl mercury; arsenic, arsenic III, and arsenic V; and chromium VI by the methods listed in Table 4-1. These test methods are not offered by USEPA-CLP and will be provided by qualified subcontract laboratories.

Methyl mercury in water and sediment will be determined by USEPA Method 1630, Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and cold vapor atomic fluorescence spectrometry (CVAFS) with sediment sample preparation by acid bromide/methyl chloride extraction. Trace total mercury in aqueous samples will be determined by USEPA method 1631. It will also be employed for sediments where it is found that the mercury detection limits offered by EPA-CLP are not sufficient to detect the presence of mercury. For all samples collected for trace metals analyses, handling methods, including field filtering, will follow the protocol provided in USEPA Method 1669.

Arsenic species (Total As, As III, and As V) in water and sediment will be measured by USEPA Method 1632 (USEPA 1998), Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry, Revision A with modification to extract the arsenic species [As(III) and As(V)] from sediments.

Hexavalent chromium will be determined in the water and sediment samples by USEPA Method SW-846-7199 employing ion chromatography. Sediment samples will be prepared by USEPA Method SW-846-3060A.

Selected sediment samples, as described in FSP Volume 1 (Malcolm Pirnie, Inc., 2005a) will also be analyzed for acid volatile sulfide – simultaneously extracted metals (AVS-SEM) which is necessary for mercury modeling. These analyses will be conducted using the extraction and analytical methods detailed in Table 4-1.

2.4.2 Organic Methods

Methods for analyses of organic parameters are listed in Table 4-2. Many of the analyses for TCL compounds in surface water and sediment samples collected during the 2005 sampling event will be performed by the methods described in the Laboratory SOW for Organic Analysis Multi-Media, Multi-Concentration, SOM01.4, October 2004 (USEPA, 2004c) or the most recent revision of the USEPA-CLP SOW. The USEPA CLP SOW provides methods for the isolation, detection, and quantitative measurement of TCL volatiles, semivolatiles, pesticides, and Aroclor target compounds in water and sediment samples. Analytical techniques such as GC-MS-Selective Ion Monitoring (GC-MS-SIM) and GC-Electron Capture Detector (GC-ECD) will be employed. In order to achieve lower RLs for selected compounds (*i.e.*, PAHs), a modification to the SOW is being requested under the CLP flexible clause.

In addition, several TCL parameters, including pesticides and PAHs, will also be analyzed by a subcontractor laboratory using the methods detailed in Table 4-2. This is due to low RL requirements, together with limited sample volumes[2].

Analyses for dioxins/furans and PCBs in sediment samples collected during the 2005 sampling event will follow a tiered analytical approach. This approach will include the use of screening and laboratory methods suited for different concentrations of contaminants. The laboratory method for determining dioxins/furans will be USEPA Method 1613B by High Resolution GC-High Resolution MS (HRGC-HRMS) which, after extraction, measures the isomers at pg/L (picograms/Liter) [parts per quadrillion (ppq)] levels in water and at ng/kg (nanograms/kilogram) [parts per trillion (ppt)] levels

2 For the high resolution coring program, limited sample volume will be available for analysis due to the diameter of the sampling device and the anticipated core segment length

in sediment samples. The laboratory analytical method for measuring PCB congeners will be USEPA Method 1668A by HRGC/HRMS. This method can attain pg/L (ppq) detection levels in water samples and ng/kg (ppt) detection levels in sediment samples.

In addition, immunoassay screening for dioxin/furans and PCBs will be conducted on select sediment samples by a modified version of USEPA Method SW-846-4025. This method is used to estimate the dioxin and PCB Toxic Equivalency Quotient (TEQ), and is available in a kit supplied by Cape Technologies. The procedure can provide a semi-quantitative estimate of both dioxin TEQ and PCB TEQ at a 20 pg/g (ppt) reporting limit on a single sediment sample. Copies of the Cape Technologies technical notes are included in Attachments 7 and 8. During the 2005 sampling event, sediment samples will be collected for analyses via both the screening method and the laboratory analytical method to evaluate the effectiveness of the screening method and if possible, to establish a correlation between the laboratory methods and the immunoassay method. This evaluation will be used to determine if the screening method can be employed to screen the large number of sediment samples to be collected in 2006.

The sediment samples will also be tested for chlorinated herbicides (USEPA Method 8151A) and total petroleum hydrocarbons (TPH) (NJDEP method Document #: OQA-QAM-025-10/91). A copy of the TPH method is included in Attachment 12. Other organic analysis test methods that will be employed are listed in Table 4-2 and include analyses of water samples for butyl tins, dissolved organic carbon (DOC), particulate organic carbon (POC), and total organic carbon (TOC).

2.4.3 Radiochemistry

Radiochemistry analyses will be conducted for sediment dating purposes and will be performed by the methods provided in Table 4-4. Radionuclides will be determined by Gamma Spectrometry and/or Alpha Spectrometry following the methods given in the Health and Safety Laboratory (HASL)-300 EML Procedures Manual and USEPA-600 4-80-032 (USEPA, 1980).

2.4.4 Other Tests and Water Quality Parameters

Methods for analyses of wet chemistry parameters are listed in Table 4-3. Water quality tests to be conducted on water column samples include total phosphate,

orthophosphate, nitrogen (ammonia and Kjeldahl), chemical oxygen demand (COD), biochemical oxygen demand (BOD), total dissolved solids (TDS), total suspended solids (TSS), volatile suspended solids (VSS), chlorophyll a, and pH.

2.4.5 Geochemistry – Engineering Tests

Geotechnical test methods are listed in Table 4-5. Low resolution core sediment samples will be tested for engineering parameters including grain size, percent moisture, Atterberg limits, and specific gravity by ASTM methods. Bulk density of the sediment cores will be field determined as described in the FSP. In addition, sediment samples will be analyzed for cation exchange capacity (CEC) by SW-846-9081.

Requested laboratory turn-around times (TATs) for the non-CLP test methods for the majority of the requested analyses will be within 35 days of receipt of the sample. Accelerated TATs may be requested for specific samples, as appropriate.

2.5 QUALITY CONTROL

To monitor the quality of the data generated for this project, an appropriate number and type of QC procedures will be employed for each measurement. The employment of QC procedures permits the validation of the method and provides a measure of the ability of the particular system being used to meet the DQOs established for each measurement or analysis. Once the measurement or analysis has begun, the employment of QC procedures permits the monitoring of the system output for quality. The QC results, presented along with the reported data, allow the data to be assessed for quality and, with other factors, allow a determination to be made on how well the data have met the DQOs. In general, laboratory QC programs are more rigorous than field QC programs. The type and frequency of the individual QC for the CLP analytical methods are given in the CLP SOW; for the non-CLP parameters this information is contained in the tables in Attachment 3.

2.5.1 Laboratory Quality Control

Both CLP and non-CLP laboratories will be required for this project. Procurement and tracking of these services will be conducted in accordance with the

memoranda below. Procurement of the non-CLP laboratories will be conducted to ensure that qualified, experienced laboratories are procured:

- Procuring Analytical Services through the DESA Laboratory and the CLP. Robert Runyon, Chief Hazardous Waste Support Section. No date.
- Tracking Superfund Non-CLP Analytical Data (ANSETS): Directive # 9240.0-2C. Jennifer Feranda, CLP Project Officer and RSCC, Hazardous Waste Support Section. No date.
- Directive # 9240.0-2C: Tracking Superfund Non-CLP Analytical data, Michael B. Cook, Director, Office of Emergency and Remedial Response. November 14, 2002.

2.5.2 CLP Laboratory Quality Control

All samples being analyzed through USEPA's CLP program (TCL organics and TAL inorganics including titanium and cyanide) will be analyzed following the QC methods described in the most recent CLP documents:

- USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration (SOM01.0), Exhibit E: Quality Assurance/Quality Control Procedure and Requirements (USEPA, 2004c). October 2004 or the latest revision.
- USEPA Contract Laboratory Program, Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration (ILM05.3), OSWER Document 9240.1-43FS, USEPA Publication 540-F-04-001. February 2004 (USEPA, 2004b), Quality Assurance/Quality Control Procedure and Requirements or the current revision.

2.5.3 Non-CLP Quality Control

For the non-CLP laboratories, a SOW was developed that lists each analytical method along with the required RLs and QC. Refer to the QC tables in Attachment 3 for the minimum non-CLP laboratory QC requirements. The SOWs in these Lab Task Orders were sent to all prospective laboratories. The laboratory bids on this work were required to demonstrate the ability to comply with these requirements. Applicable QC requirements are included in Attachment 3.

Subcontracting with the non-CLP laboratories was a major acquisition, which is described in the Final QCP (Malcolm Pirnie, Inc., 2003b) as requiring detailed source selection decision-making criteria. As such, prior to selecting any subcontract laboratories, certain minimum requirements had to be be met. Each laboratory was be

selected based on an objective, qualifications-based evaluation prepared by Malcolm Pirnie. The qualifications included in this evaluation included, but are not limited to, the following:

- Documentation that the laboratory has the appropriate certifications/accreditations.
- An initial demonstration of capability is required from all laboratories for all applicable methods prior to analyzing environmental samples.
- Documentation that the laboratory has met the analytical method's specific performance criteria requirements.
- Documentation that the laboratory has conducted a determination of the method detection limit, as described by the analytical method and where appropriate.
- Each analyst must have completed a demonstration of capability prior to analyzing environmental samples. If modifications are made to a method protocol which could change detection limits, the initial demonstration of capability must be repeated.
- Each laboratory must maintain a formal in-house QA/QC program to which they adhere.
- Each laboratory must demonstrate that they adhere to their own SOPs.
- The laboratory must demonstrate that that are able to meet the sample capacity and turn around time requirements.

Malcolm Pirnie will monitor to determine that the laboratories are in compliance with the SOWs through the data validation process (refer to Section 4 – Data Validation and Usability, of this QAPP).

2.5.4 Special QC Requirements for Organic Water Column Samples

For the 2005 water column sampling events, various methodologies will be used to collect samples in order to evaluate the most appropriate methodology that should be used for the overall water column program. These samples will use the following QC requirements, with the exceptions/additions detailed in Sections 2.5.4.1 and 2.5.4.2.

- Rinsate (Equipment) Blanks will be collected at a rate of one per week of sampling from each lot of decontaminated or dedicated equipment.
- Field Duplicates will be collected for grab samples at a rate of 1 field duplicate for every 20 samples.
- Matrix Spike/Spike Duplicate Samples will be collected for grab samples at a rate of 1 pair for every 20 samples.

2.5.4.1 Semi-Permeable Membrane Device (SPMD)

- Lipid Blank - Analysis of lipid used in SPMD for organic analytes.
- Field Duplicate - One field duplicate collected from SPMD in brackish water and one field duplicate collected from SPMD in fresh water.
- Field Blank – One field blank (*i.e.*, non-deployed, but opened SPMD taken to sample locations) collected during deployment and one field blank collected during retrieval of SPMDs.

2.5.4.2 Infiltrex or Other Large Volume Sampling Device

- XAD™ resin column Labeled Surrogates will be used to measure recovery of analytes and any wash out that may have occurred.
- Lab Method Blank on XAD™ resin traps and filters.
- Field Blank – A least one field blank will be collected for both the XAD™ trap and the glass filter. The XAD™ field blank will be collected by leaving the ends of the column open, while the filled column is being loaded into the sampler. Similarly a glass fiber filter blank will be collected by exposing a filter to air while loading the sample filters.
- Parallel whole water sample to check for variability; this sample will be collected as a series of grab samples collected over the same time period as the Infiltrex, which are then composited for analysis.

2.6 PREVENTATIVE MAINTENANCE AND INSTRUMENT CALIBRATION

When collecting field measurements or analyzing data, only calibrated instruments will be used. Instruments must be properly calibrated to produce technically valid data. Documentation of calibration and response check results verifies that the instruments used for measurement are in proper working order and the data produced are reliable. The calibration requirements described below are necessary to support the DQOs for this project. Calibration of field instruments will be documented in the field laptop and uploaded to PREmis.

The purpose of a preventative maintenance program is to keep the calibrated sampling, field testing, and analytical equipment working properly, confirm proper performance, avoid erroneous results, and minimize equipment downtime. The preventative maintenance program also provides for the documentation of all maintenance to be used as evidence of instrument maintenance and for scheduling future

maintenance. The laboratory preventative maintenance program is the responsibility of the laboratory and only the minimum requirements are mentioned here.

2.6.1 Field Instruments

As described in the FSP, various instruments will be utilized to collect measurements in the field. To confirm that equipment is working properly, and to avoid erroneous results, these instruments will be maintained under the preventative maintenance program described below:

- On at least an annual basis (if applicable), equipment will be calibrated by the manufacturer or other qualified facility. The calibration records will be maintained in the site files.
- At a minimum, instruments will have a battery and response check at the start of each day, before measurements are made, and at the end of each day, after all measurements are complete. Any response checks conducted by the field crew will be recorded in the field laptop and uploaded to PREmis. If the initial response check indicates a problem with the instrument, it will not be used in the field until the problem is corrected. If the end of the day response check indicates a problem with the instrument, the preceding sample results will be reviewed for validity and reanalyzed as necessary. Field calibration will be conducted at the interval recommended by the manufacturer.
- Minor service and repair will be done by the Equipment Manager, who is trained in the service and repair of field instruments. Equipment in need of major or more complex repair and services will be sent to the manufacturer or other qualified facility. All maintenance, servicing, and repair will be recorded and kept on file. Field personnel will record maintenance and instrument problems in the field laptop. The Equipment Manager will keep a record of all equipment released to the field and a record of all maintenance and service on file.
- Normal upkeep will be conducted daily after each use and includes inspecting for damage and signs of problems and will include, as appropriate:
  - Cleaning.
  - Lubrication of moving parts.
  - Check/change battery.
  - Inspect for damage.
  - Check for operation problems.
  - Inspect all hoses and lines.
- Information to be recorded during a field calibration or response check could include, as applicable, date and time, technician name, field calibration or response check procedure, response check results, problems, and instrument serial numbers.

- All calibration standards will be traceable to acceptable sources. Only personnel trained in the use of the field instruments will operate them.

The specific operation and maintenance of the field equipment to be used during the project is documented in the FSP. Note that the operation and maintenance program for the mooring equipment (Hydrodynamic/Sediment Transport Program) is different than the program outlined above (refer to Attachment 4 to FSP Volume 1). The manufacturer's suggested maintenance program for the equipment is specified in the FSP.

If any of the equipment used for this project is rental equipment, it must be demonstrated that the rented equipment will be able to meet the DQOs of the data collection activity for which the equipment is being used. As a result, the equipment supplier will be required to provide adequate documentation of the accuracy, maintenance, and upkeep of the rented equipment that will enable the DQOs to be met.

2.6.2 Laboratory Instruments

The primary goal of the project laboratories' preventive maintenance programs will be to prevent instrument and equipment failure as much as possible and to minimize instrument downtime when failures occur. The laboratories selected will maintain an inventory of replacement parts needed for preventative maintenance and spare parts that routinely need replacement. Implementation and documentation of the preventive maintenance program will be the responsibility of the technical group using the instrument according to the individual policies in the Laboratory Quality Manual. If an instrument failure impedes sample analysis, the laboratory will notify the SQO of the problem so corrective actions can be implemented, including sample capacity management.

2.7 LABORATORY INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

All samples collected for this project will be analyzed according to specific USEPA or other established procedures. The preventative maintenance and calibration procedures and frequencies for these analyses are detailed in each applicable analytical method. All calibration results will be received from the laboratory as part of the data package deliverable and they will be kept in the site file and verified as part of the data

validation process. For the non-CLP laboratories, additional calibration information is referenced in Attachment 3. The preventative maintenance activities, either preventative or repair, will be documented on standard forms or logbooks. Written procedures will include maintenance schedules, problem identification procedures, space for describing problems and repair notes, and failure analysis protocols. Service contracts and regularly scheduled in-house maintenance will be included, along with a list of critical spare parts. In the event a piece of equipment breaks down for an extended period of time, the laboratory will have sufficient backup equipment to complete the analyses within holding time requirements.

2.8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

All supplies and consumables used for this investigation will be obtained through appropriate suppliers and will meet any applicable supply-specific requirements. All supplies and consumables will be inspected prior to use. Any product that does not meet applicable requirements will be returned to the supplier for replacement or will be discarded. Supply-specific requirements include, but are not limited to, the following:

- Blank water will be certified analyte-free and analytical results will be provided for each lot.
- Decontamination and preservation chemicals will be ultra-pure grade or pesticide-grade, as applicable. Certifications will be obtained from the supplier.
- Sampling equipment will be constructed of approved materials.

2.9 NON-DIRECT MEASUREMENTS

There are several non-direct measurements that will be used during the investigation. These include historical data for various media, atmospheric deposition measurements, hydrodynamic studies, and fresh water inflows. The details regarding the evaluation of these measurements and how they will be used are described in detail in the WP (Malcolm Pirnie, Inc., 2005c) and FSP Volume 1 (Malcolm Pirnie, Inc., 2005a).

2.10 DATA MANAGEMENT

This section describes the project data management process, tracing the path of the data from their generation to their final use or storage.

2.10.1 Field Data

Due to the magnitude and complexity of the sampling program, traditional field data collection methods (*e.g.*, handwritten field logbooks and data sheets) are impractical for this project. Therefore, PREmis, a centralized, web-based data management system, has been created. Data collection occurs on a web-based application (developed in-house at Malcolm Pirnie) accessed through a field laptop computer. As the field team inputs information into the laptop, this information is transmitted via wireless technology to the project website. Note that if the wireless connection is lost, the field application will store the information locally until the connection is reestablished. Refer to Attachment 9 for a memorandum describing security procedures for the field application. Once on the project website, the data are available to project team members in the following formats such as:

- A Microsoft Access or Excel download.
- A report available for viewing on the website.
- On the live GIS map available on the website.
- A pdf download for field notes or sketches.
- A thumbnail or download for digital site photographs.

The following section summarizes data collection from the field to the project website:

- First, a secure project website is established; this website is PREmis. Security on the website consists of secure socket layers (*i.e.*, https site), password protection, and multiple user levels. These user levels restrict access and rights to certain portions of the website.
- A calendar of field events (with a comments section) is created to assist the field team(s) with their work, and to ensure that all teams know and understand their sampling assignments. Work orders that specify where sampling is to occur, what parameters should be analyzed for, as well as any other pertinent information, are also created in the calendar.

- When the field team(s) begins work, each team is assigned a field laptop that has a specific identification number associated with it. When the field team launches the field application, the user is prompted for their unique username and password. This way, the field application keeps a log of who entered information, along with the dates and times the information was entered. The purpose of this is twofold: this acts as each field team member's electronic signature and it also ensures that unauthorized users cannot access the software (*i.e.*, write in someone else's logbook).
- At the beginning of each new sampling event, the field team creates sampling stations for sample locations that are specific to that field team.
- Instrument QC is entered directly into the system at the beginning and end of each day, as applicable. If the response check indicates that the instrument is not working properly (*e.g.*, the photoionization detector (PID) response is greater than 2 ppm different from the standard gas concentration), the user is prompted to use a different instrument. This allows the field team to immediately identify if a problem is occurring, thus eliminating wasted field effort.
- When the field team begins collecting sampling information, they are required to fill in a series of information windows that consist of pick lists, comment fields, and automatically generated fields. For example, if a field team is collecting a chemical sediment sample, the field application, *not the field team*, assigns the sample ID and also creates the sample label. Since the sample ID also contains the unique identifier for the laptop from which it was requested, sample IDs are never duplicated. Another advantage is the elimination of missing information since certain fields must be filled in prior to moving to another window.
- As the field team collects field measurements and laboratory samples, the field application prompts them to collect QC samples (*e.g.*, duplicates, triplicates, MS/MSD, MS/MD, rinsates). Certain QC calculations for field measurements are built into the system. For example, when the field team collects a duplicate measurement with an instrument, the field application will calculate the RPD and determine if it falls within the required limits. If not, a message will appear on the screen warning the user to check the instrument.
- After the field team completes an information window and clicks the button labeled "Done," the information entered into the window can be viewed but it cannot be changed. This is analogous to the field team not being allowed to erase information once it's entered into the field logbook.
- All the information collected in this application is written to a secure password-protected Microsoft Access database accessible directly only by a database administrator. Since the database is secure, the field team is not able to make any changes to the records contained in it.
- Since the field application uses wireless technology, all information entered into the application is automatically uploaded to the project website. If there are any problems with the wireless system, the information is stored in the laptop until the field team returns to the field office to upload the information to the project website. The field team prints out the field data collection report from the website, reviews the

report, and initials and dates each page. Copies of this report are kept at the site field office under the field team leader's control

- Once the information is on the website, it is reviewed by the SQO or his designee. They can either accept or reject each piece of data. Until the SQO marks the data as reviewed and either approved, conditionally approved, or rejected, only personnel with the proper security level can view the data. The data can be viewed by the entire project team only after the SQO review is complete.
- During the SQO review and/or the field team's review of the report, it is possible that mistakes or omissions in the information will be noted. When this occurs, the field team is supplied with a paper form to fill out that requests either supplemental information or corrections to the data. This information is then added to the report by one of the site administrators. A complete paper record of the change and/or addition, the person requesting the correction, the person supplying the information, and the date of the change, is maintained in the site files.
- As described above, once the field data are collected, the information is uploaded from the field application to the project website. A module on the website allows the field team to select individual samples, create chain of custody forms, and mark the samples as shipped to the laboratory. Each chain of custody form is retained electronically on the system; a signed hard copy of the form is also retained in the site files, under control of the field team leader.

2.10.2 Laboratory Data

As described above, data collected for this project will be stored electronically in PREmis. The following describes the process for managing data from the laboratory:

1. Once the field information is uploaded to PREmis and approved by the SQO or designee, laboratory samples will appear on the data report. Prior to receiving data from the laboratory, these samples will be marked to indicate that laboratory data is outstanding.
2. All samples will be sent to the laboratory following the COC procedures detailed in this QAPP. Once the laboratory receives the samples, a module on the website allows them to mark each shipment as received. Any problems with the shipment such as broken custody seals or insufficient sample volume are also marked on the website. Note that CLP laboratories will not be required to fill out the information on the website. They will supply RSCC with a sample receipt checklist; Malcolm Pirnie will enter this information into the website.
3. The laboratories used for this project will utilize USEPA CLP or equivalent sample handling procedures. Each laboratory utilized for this project will be required to have a laboratory information management system (LIMS) capable of producing EDDs.
4. When the laboratory analyzes the samples, raw data is generated. This data, which can take the form of area counts or instrument responses, is processed by the laboratory as described in the analytical method, and converted into concentrations.

5. The laboratory then generates an EDD that contains a variety of information including, but not limited to the following [Note that the CLP laboratories will create a USEPA Multimedia EDD (MEDD) while the non-CLP laboratories will create an MEDD equivalent EDD.]:
   - Sample ID.
   - Chemical Abstracts Services (CAS) Number.
   - Preparation Method.
   - Analytical Method.
   - Cleanup Method.
   - Collection, Preparation, and Analysis Date.
   - Dilution Factor.
   - Percent Moisture.
   - Analyst Name.
   - Instrument ID.
   - Concentration.
   - RL and DL.
   - Laboratory Qualifier(s).
   - Unit.
6. The EDD is uploaded directly to PREmis through a module on the website. The CLP MEDD will be uploaded by Malcolm Pirnie while the non-CLP EDD will be uploaded by the subcontract laboratory.
7. Once this information is uploaded, only personnel with the proper security level can view the data. First, the data must be validated (see Section 4 – Data Validation and Usability, of this QAPP) and the validator makes changes directly to the data stored in the website (*e.g.*, add validation qualifiers, change concentrations based on blank data). Any changes made to information contained in PREmis is recorded in an electronic audit record; this record stores the original value, the changed value, the name of the person who made the change, and the date and time of the change. Next, the SQO or designee reviews and approves or reviews and changes any changes made by the validator. Once these changes are approved, the data can be viewed by the entire project team.
8. Since all of the data are collected electronically, and since the QC samples are automatically associated with each original sample, the system also generates sample trip reports for use by the data validator.

3.0 ASSESSMENT AND OVERSIGHT

This element addresses assessment of the effectiveness of the project implementation and associated QA/QC activities.

3.1 ASSESSMENT AND RESPONSE ACTIONS

To monitor the capability and performance of the FSP activities, several types of audits will be performed. TSAs are field audits that monitor the field techniques, procedures, and overall implementation of the WP (Malcolm Pirnie, Inc., 2005c), FSP, and QAPP. These audits will be conducted by the SQO or designee. Performance audits (PAs) of laboratories are conducted to measure the accuracy of the measurement systems. Data Quality Audits (DQAs) are conducted to determine if the data generated by the sampling and analysis satisfy the DQOs.

3.1.1 Technical System Audits (TSAs)

Field audits will be conducted on an ongoing basis during the project as field data are generated, reduced, and analyzed. Numerical manipulations, including manual calculations, will be documented in a field logbook. Records of numerical analyses will be legible, of reproduction-quality, and sufficiently complete to permit logical reconstruction by a qualified individual other than the originator.

System audits of site activities will be accomplished by an inspection of field site activities. During this audit, the auditor(s) will compare current field practices with standard procedures. The following elements will be evaluated during a TSA:

- Whether activities are conducted in accordance with the WP (Malcolm Pirnie, Inc., 2005c).
- Whether procedures and analyses are conducted according to procedures outlined in the FSP.
- Whether proper sample documentation is being recorded.
- If the working order of instruments and equipment is being properly checked and recorded.
- The level of QA conducted per each field team.

- Contingency plans in case of equipment failure or other event preventing the planned activity from proceeding.
- Decontamination procedures, if applicable.
- Level of efficiency with which each team conducts planned activities at one site and proceeds to the next.
- Sample packaging and shipment.

TSAs are conducted for each field team at the beginning of each field sampling task to determine if the system is capable of producing data that meet the DQOs. As long as the field team(s) demonstrate proficiency in the sampling procedures being audited, a follow-up audit will not be required. However, if the audit indicates the need for corrective action, a second TSA will be required. Following the initial audit, TSAs will be conducted on the following schedule:

- Whenever key personnel leave the project or new key personnel are added to the project.
- Whenever a significant amount of time (more than 6 months) has elapsed between TSAs for a particular field task.

Any minor deficiencies that are noted during the TSA will be corrected in the field as they occur. If major deficiencies are noted (*i.e.*, those that cannot be immediately corrected in the field), a Stop-Work Order will be issued until appropriate measures can be taken to correct the problem. A Stop-Work Order may be issued by the SQO, following notification to the PM. The conditions and the need for a Stop-Work Order will be documented in sufficient detail to permit evaluation of the deficiency and determination of proper corrective action(s). Pertinent communications with the Field Team Leader, SQO, DPM, and PM that pertain to an evaluation of the problem along with potential solutions and their implementation will be attached to the Stop-Work Order. In order for work to resume following a Stop-Work Order, the Malcolm Pirnie PM and SQO must rescind it in writing. The SQO is responsible for tracking non-conforming conditions, evaluating the effectiveness of corrective measures, and assuring that the necessary steps have been taken to prevent recurrence of the original problem.

Regardless of whether major, minor, or no deficiencies were noted during the audit, a written report of the TSA will be prepared by the SQO and submitted to the

Malcolm Pirnie, USEPA, and USACE PMs, as well as the Field Team Leader and the field team. This report will identify any deficiencies found and will outline the corrective actions that were recommended/implemented to address them. A copy of SOP No. 3 on conducting a TSA and an example of an audit form are found in Attachment 10. Note that the audit form contained in the SOP is for example purposes only; the SQO will tailor this form for each type of activity audited. Periodically during the audit, it may be determined that the site program should be modified to increase data quality or efficiency. These modifications will be documented by the Malcolm Pirnie PM or SQO in a Field Modification Form. An example of this form can be found in Attachment 11.

3.1.2 Field Corrective Actions

At the end of each sampling day, the sampling team is to report any problems requiring corrective action that were encountered during the day. Corrective action will be undertaken when a non-conforming condition is identified. A non-conforming condition occurs when QA objectives for precision, accuracy, completeness, representativeness, or comparability are not met, or when procedural practices or other conditions are not acceptable. A report is to be filed that documents the problems encountered and the corrective action implemented. A Stop-Work Order may be issued by the SQO, following notification to the PM, if corrective action does not adequately address a problem, or if no resolution can be reached.

3.1.3 Performance Audits

A PA consists of sending a laboratory a performance evaluation (PE) sample for analysis. The PE sample is a sample of known concentration [established by an independent party such as the National Institute of Standards and Technology (NIST)] that is analyzed by the laboratory and the analytical results are compared with the certified concentration. The results provide a measure of laboratory performance that is used along with other QA criteria to monitor laboratory capability. At the current time, there are no plans to conduct a PA. Therefore, all chemical subcontract laboratories procured for this project must be NELAC certified and are subject to the performance audits required by that program.

3.1.4 Internal Laboratory Audits

As part of its QA program, the Laboratory Quality Assurance Manager (QAM) will conduct periodic checks and audits of the analytical systems to ensure that the systems are working properly and personnel are adhering to established procedures and documenting the required information. These checks and audits will also assist in determining or detecting where problems are occurring.

In addition to conducting internal reviews and audits, as part of its established QA program, the laboratory is required to take part in regularly scheduled Performance Evaluations and laboratory audits from State and Federal agencies for applicable tests. Each laboratory selected to support this program must maintain current State and Federal certifications, as appropriate.

3.1.5 Laboratory Corrective Actions

If a particular laboratory analysis is deemed "out of control", corrective action will be taken by the laboratory to maintain continued data quality. Each laboratory must adhere to their in-house corrective action policy. The coordinator of the laboratory's analytical section will be responsible for initiating laboratory corrective action when necessary.

3.1.6 Data Quality Audits (DQAs)

DQAs are conducted to determine if the data are adequate to support the DQOs and to determine the cause of deficiencies in the event that the data quality is not adequate. This audit is conducted by the SQO after the data have been fully validated. The SQO will first determine to what extent the data can be used to support the decision making process. If the data are deficient, the SQO will identify the cause of the deficiency and will determine what modifications need to be made (*e.g.*, have the laboratory analyze a larger volume sample to lower the RLs) so that subsequent data are acceptable.

3.2 REPORTS TO MANAGEMENT

The USACE and USEPA PMs will receive several types of management reports. These will include the results of any TSAs, corrective action reports, and data validation/usability reports. In addition, the monthly progress report will contain a section on quality control reports. Problems or issues that arise between regular reporting periods may be identified to program management at any time. Information included in the progress report will include the following:

- Results of Technical System field audits conducted during the period;
- An assessment of any problems with the measurement data, including accuracy, precision, completeness, representativeness, and comparability;
- A listing of the non-conformance reports including stop-work orders issued during the period, related corrective actions undertaken, and an assessment of the results of these actions; and
- Identification of significant quality assurance problems and recommended solutions, as necessary.

4.0 DATA VALIDATION AND USABILITY

Date Validation and Usability are implemented so that the individual data elements conform to the specified criteria and to enable reconciliation with the project's objectives. This group of elements covers the QA activities that occur subsequent to the data collection phase of the project.

4.1 DATA REVIEW, VERIFICATION AND VALIDATION

4.1.1 USEPA CLP Data

Validation will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in the applicable analytical methods and the laboratory SOWs. A sample trip report will be generated by PREmis that correlates QA/QC samples (*e.g.*, rinsate blanks, duplicates) with their associated environmental samples. All TAL/TCL data generated through the CLP will be validated by RSCC using the latest applicable USEPA Region 2 validation procedures and according to the following USEPA national guidance documents or their most recent revisions:

- USEPA CLP National Functional Guidelines for Organic Data Review, OSWER 9240.1-5A-P, October 1999.
- USEPA CLP National Function Guidelines for Inorganic Data Review, OSWER 9240.1-45, October 2004.

4.1.2 USEPA DESA Laboratory Data

Data generated by the USEPA Region 2 DESA laboratory in Edison, NJ are considered USEPA-validated and are useable as reported. No third party data validation will be performed on DESA-generated data.

4.1.3 Subcontractor Laboratory Data

The subcontractor laboratory data will be validated by Malcolm Pirnie or a qualified subcontractor. The dioxin/furan data will be validated in accordance with

USEPA Analytical Operations/Data Quality Center (AOC) National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, OSWER 9240.1-37, August 2002. The remaining parameters will be validated in accordance with the QC requirements in Attachment 3, USEPA's National Functional Guidelines and applicable Region 2 guidelines.

For dioxins/furans and PCB congeners, a 100% validation will be conducted for each data package. For all other parameters, the validator will conduct a 100% validation for the first two sample delivery groups (SDGs) received for each analytical parameter. This means that the validator will review the raw data and log book sheets, and will recalculate at least 10 percent of the sample and QC sample results. If this validation indicates that the laboratory is producing acceptable data, the validation will be scaled back and subsequent data packages will have a less rigorous review. The validation will then be based on the information provided by the laboratory on their QC forms. If the laboratory QC on the report forms are within limits no further review will be conducted. However, if there are QA/QC aspects not meeting criteria, the validator may then review some or all of the full data package to determine the cause or data quality impact of the non-compliance. In addition, at least one of every ten data packages will be subject to a full review.

Once data validation is completed, a data validation report will be generated. The report will contain information regarding the parameters that are qualified, the reason for the qualification, and the direction of the bias (only for parameters qualified as estimated). The validation report will be uploaded to the Digital Library in PREmis and the validation qualifiers will be added to the electronic data stored in PREmis.

Based upon the quality assurance review of the analytical data, specific codes (data qualifiers or 'flags') will be placed next to results in the database to provide an indication of the quantitative and qualitative reliability of the results. The following data qualifier codes are proposed for this project:

- U: The compound/analyte was analyzed for, but was not detected above the sample RL. This applies to both samples in which the sample was reported as not detected by the laboratory, as well as compound/analytes which are considered "not detected" (*i.e.*, negated by the data validator) due to their detection in a blank at a similar level, as determined during the data quality review/data validation process.

- E: Quantitation is approximate (estimated) due to limitations identified during the quality assurance review (data validation). This qualifier is applied to all data which were reported as detected at a concentration outside the limits of the calibrated range of the analysis, as well as for other reasons (minor deviations from QA/QC criteria) as determined during the data quality review/data validation process.
- R: Unusable (rejected) result – compound/analyte may or may not be present in this sample.
- N: There is presumptive evidence to make a tentative identification of the compound
- UE: This compound/analyte was not detected, but the quantitation/detection limit is uncertain due to QA/QC issues identified during the quality assurance review.

Note that the qualifiers detailed above are only those used by the data validator. Additional laboratory qualifiers will be present on the data and will be assigned by the laboratory. CLP qualifiers are detailed in the appropriate SOWs. Qualifiers assigned by non-CLP laboratories will be defined by each laboratory in their data package and will be included in the meta-data on PREmis.

4.1.4 Field Data Evaluation

Procedures to evaluate field data for this program include reviewing the data entered into the field laptop computers to insure that errors have not been made. The field data documented includes data generated during measurement of field parameters, observations, results of any quality control sample analyses, and field instrument calibrations. This task will be the responsibility of the SQO or designee.

4.2 VERIFICATION AND VALIDATION METHODS

This section describes the process for verifying (*i.e.*, determining that project data were collected in a way that meets at least the specified QC acceptance criteria) and validating (*i.e.*, determining that the project results are suitable for use in making the specified decisions) project data. The data verification and validation steps are described below.

4.2.1 Data Verification

- Data verification begins in the field during field data entry. Instead of field logbooks, data entry is conducted via a laptop field application developed for this project. Since the application is project-specific, required fields and task-tailored data entry fields nearly eliminate missing data. In addition, validation of key fields is conducted by the application (*e.g.*, when northing and easting information is entered it is validated against coordinate limits for the study area. If the coordinates fall outside the study area, the user is prompted that the information may be incorrect), which greatly reduces the amount of accidental transcription errors.
- The field application also conducts the majority of required calculations through built-in macros so that manual calculations are not required (*e.g.*, the weights of the cores are entered into the system and the system calculates the bulk density).
- Once the field crew finishes collecting information for the day the Field Team Leader or designee is required to review the data for errors or omissions. If any are found, the project website has a field where errors/omissions are described. This information is sent to the SQO or designee for correction.
- In addition, the SQO or designee is responsible for reviewing field data for completeness and to verify that the field crew followed the QC requirements detailed in this QAPP (*e.g.*, the collection of QC samples at the required frequency, response checking the field instruments). If any problems with the information are found, the SQO or designee will document the problems on the project website. There is also a location on the website where changes to the collected information can be made. Only personnel with the required security level can access this module. All changes to information stored in the database are recorded in an audit table that records the person making the change, the original entry, and the date/time of the change.
- Once the SQO or designee reviews the field data, there is a section on the project website where the data can be marked as reviewed and either approved, conditionally approved, or rejected.
- As soon as the data are marked as reviewed by the SQO or designee they are available on PREmis to view, map, or download. Since the data are in an electronic format as they are collected, there are no manual transcription errors.
- To further reduce transcription errors, data are obtained from CLP and subcontract laboratories in an EDD created by the laboratory's LIMS. The EDD is uploaded directly into the website without the need for any manual data entry.

4.2.2 Data Validation

As described in Section 4.1 all laboratory data collected for this project will undergo validation. The following steps are involved in the data validation process:

- As environmental samples are collected, associated QC samples (*e.g.*, field duplicates, rinsates) are noted in the field application. This information is used to generate a sample trip report, available on PREmis.
- The data collected in the field application are also used to generate electronic sample labels and COC forms, thereby minimizing transcription errors and preserving sample chain of custody.
- As mentioned above, once data are received from the laboratory, the EDD is uploaded to PREmis; CAS numbers are used to identify the analytical parameters.
- The data validator validates the data in accordance with the protocols outlined in Section 4.1. As part of the data validation process, the validator identifies any qualifications, the bias, if known, of the data, and the usability of the data. The validator applies validator and bias qualifiers to the data stored in PREmis and uploads all validation reports to the Digital Library on PREmis.
- Once the validation package is received from the validator it is reviewed by the SQO or designee. Any problems with the validation will be discussed with the validator and resolved. The SQO or designee will then mark the data as being reviewed and approved. Until the data are marked as reviewed by the SQO or designee only team members with the required security level can access the data (typically QC team members). This prevents the release of unvalidated data.
- Since the data are stored electronically, a check can then be made to determine whether the completeness of the data is acceptable.
- The data users will use the data validation information when performing data evaluation and using the data.

4.3 RECONCILIATION WITH USER REQUIREMENTS

The SQO, in conjunction with the PM, will determine whether field and analytical data or data sets meet the requirements necessary for decision-making. The results of the measurements will be compared to the DQOs set forth in Attachment 1.1 of the QAPP. As data are evaluated, anomalies in the data or data gaps may become apparent to the data users. Data that do not meet the DQOs will be identified and appropriately noted in the project database so data users are aware of any limitations or concerns with the usability of the data.

If systematic problems with the laboratory data are encountered, the SQO will review the data to determine whether problems are field- or laboratory-related. The laboratory will be contacted for their analysis of the situation, along with

recommendations to correct the problem. If the problem persists, a new subcontract laboratory may be required.

5.0 REFERENCES

American Public Health Association, 1998. Standard Methods for the Examination of Water and Wastewater, 20th Edition. Prepared and published jointly by American Public Health Association, American Water Works Association, Water Environment Federation. Edited by L.S. Clesceri, A.E. Greenberg, and A.D. Eaton. Published at the American Public Health Association, Washington, DC. 1998.

ASTM, 2004. Annual Book of ASTM Standards, Section Four, Soil and Rock. 2004.

ASTM, 2004. Annual Book of ASTM Standards, Section Eleven, Water and Environmental Technology. 2004.

Battelle, 2005. Pathways Analysis Report. Prepared under contract to Malcolm Pirnie, Inc. for the USEPA and USACE. August 2005.

Cape Technologies, 2005. Technical Note TN005: Preparation of Samples for PCB-TEQ Analysis Using Carbon Column Fractionation. March 2005.

Cape Technologies, 2004. Technical Notes TN-004: Quantitation, Calibration, and Quality Assurance for Method 4025m. October 2004.

Malcolm Pirnie, Inc., 2005a. Lower Passaic River Restoration Project Field Sampling Plan: Volume 1. Draft, April 2005.

Malcolm Pirnie, Inc., 2005b. Lower Passaic River Restoration Project Field Sampling Plan: Volume 3. Revised Preliminary Draft, July 2005.

Malcolm Pirnie, Inc., 2005c. Lower Passaic River Restoration Project Work Plan. August 2005.

Malcolm Pirnie, Inc., 2005e. Lower Passaic River Restoration Project Health and Safety Plan. January 2005.

Malcolm Pirnie, Inc., 2003a. Final Passaic River Estuary Pre-Expansion Activity Plan. Section 4.7, Web Site and GIS System. February 2003.

Malcolm Pirnie, Inc., 2003b. Final Quality Control Plan. Section 6.0, Subcontractor Management and Control. February 2003.

NOAA, 2004. Extraction, Cleanup, and Gas Chromatographic/Mass Spectrometry Analysis of Sediments and Tissues for Organic Contaminants, NOAA Technical Memorandum NMFS-NWFSC-59. March 2004.

USACE, 2001. USACE Requirements for the Preparation of Sampling and Analysis Plans. 2001.

USDOE, 1997. HASL-300 EML Procedures Manual, U.S. Department of Energy, 28th Edition, Volume 1. February 1997.

USEPA, 2005. Innovation Technology Verification Report, Technologies for Monitoring and Measurement of Dioxin and Dioxin-like Compounds in Soil and Sediment, Draft Version 4. January 2005.

USEPA, 2004a. Contract Laboratory Program Guidance for Field Samplers. 2004.

USEPA, 2004b. USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration (ILM05.3). OSWER Document 9240.1-43FS, USEPA Publication 540-F-04-001. February 2004.

USEPA, 2004c. USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration (SOM01.0). October 2004.

USEPA, 2002a. Multi-Media Dioxin and Furan Analytical Service for Superfund (DLM01.4), OSWER Document 9240.1-38FS USEPA Publication 540-F-02-007, September 2002.

USEPA, 2002b. Guidance on Environmental Data Verification and Data Validation, USEPA QA/G-8, November 2002.

USEPA, 2002c. Guidance for Quality Assurance Project Plans. USEPA QA/G-5. December 2002.

USEPA, 2001. Requirements for Quality Assurance Project Plans. USEPA QA/G-5. March 2001.

USEPA, 2001. Method 1630 Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and CVAFS, Draft January 2001.

USEPA, 2000. Guidance for the Data Quality Objectives Process, QA/G-4. August 2000.

USEPA, 1999. Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/MRMS. EPA-821-R-00-002. December 1999.

USEPA, 1998. Method 1632 Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry, Revision A. August 1998.

USEPA, 1997. Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Matrices, 2nd Edition. September 1997.

USEPA, 1996. SW-846, "Test Methods for Evaluating Solid Waste," including Promulgated Final Update III. 3rd Edition. December 1996.

USEPA, 1996. Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels. July 1996.

USEPA, 1994. Method 1613, Revision B: Tetra through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. October 1994.

USEPA, 1991. Draft Analytical Method for the Determination of Acid Volatile Sulfide and Selected Simultaneously Extractable Metals in Sediment. EPA-821-R-91-100. December 1991.

USEPA, 1983. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020. Revised March 1983.

USEPA, 1980. Prescribed Procedures for Measurement of Radioactivity in Drinking Water. USEPA-600 4-80-032. August 1980.

USEPA Region 1, 2002. Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, and Ethene, Revision 1. February 21, 2002.

USEPA Region 2, 1988. Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method). July 27, 1988.

USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration (DLM01.4).

USEPA Method 440.0, Determination of Carbon and Nitrogen in Sediments and Particulates of Estuarine/Coastal Waters Using Elemental Analysis.

6.0 GLOSSARY AND ACRONYMS

%R	Percent Recovery
ADCP	Acoustic Doppler Current Profiler
AES	Atomic Emission Spectroscopy
AL	Action Level
AOC	Analytical Operations/Data Quality Center
ARAR	Applicable or Relevant and Appropriate Requirement
ASTM	ASTM is the name of the non-profit standards organization formerly called the American Society for Testing and Materials
AVS	Acid Volatile Sulfide
Be-7	Beryllium-7
BOD	Biological Oxygen Demand
CARP	Contaminant Assessment and Reduction Program
CAS	Chemical Abstracts Services
CEC	Cation Exchange Capacity
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CIH	Certified Industrial Hygienist
CLP	Contract Laboratory Program
COC	Chain of Custody
COD	Chemical Oxygen Demand
COPC	Chemical of Potential Concern
COPEC	Chemical of Potential Ecological Concern
Cs-137	Cesium-137
CSM	Conceptual Site Model
CSO	Combined Sewer Overflow
CSP	Certified Safety Professional
CTD	Conductivity, Temperature, and Depth
CVAA	Cold Vapor Atomic Absorption
CVAFS	Cold Vapor Atomic Fluorescence Spectrometry
DEFT	Decision Error Feasibility Trials
DESA	Division of Environmental Science and Assessment
DL	Detection Limit
DOC	Dissolved Organic Carbon
DOT	Department of Transportation
DPM	Deputy Project Manager
DQA	Data Quality Audit
DQO	Data Quality Objectives

ECD	Electronic Capture Detector
EDD	Electronic Data Deliverable
EIS	Environmental Impact Statement
EML	Estimated Method Limit
EMPC	Estimated Maximum Possible Concentration
EPC	Exposure Point Concentration
ERRD	Emergency and Remedial Response Division
FS	Feasibility Study
FSP	Field Sampling Plan
GC	Gas Chromatography
GC-ECD	Gas Chromatography-Electron Capture Detector
GC-FPD	Gas Chromatography-Flame Photometric Detector
GC-MS	Gas Chromatography-Mass Spectrometry
GC-MS-SIM	Gas Chromatography-Mass Spectrometry-Selective Ion Monitoring
GIS	Geographical Information System
HASL	Health and Safety Laboratory
HASP	Health and Safety Plan
HI	Hazard Index
HOC	Hydrophobic Organic Compound
HRGC/HRMS	High Resolution Gas Chromatography-High Resolution Mass Spectrometry
HRGC/LRMS	High Resolution Gas Chromatography-Low Resolution Mass Spectrometry
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectrometry
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
LCS	Laboratory Control Standard
LIMS	Laboratory Information Management System
LISST	Laser In-Situ Scattering and Transmissometry
MD	Matrix Duplicate
MDL	Method Detection Limit
MEDD	Multi-Media Electronic Data Deliverable
MS	Mass Spectrometer *or* Matrix Spike
MSD	Matrix Spike Duplicate
NCP	National Contingency Plan
NELAP	National Environmental Laboratory Accreditation Program
NEPA	National Environmental Policy Act
ng/kg	nanogram/kilogram

NIST	National Institute of Standards and Technology
NJDEP	New Jersey Department of Environmental Protection
NJDOT	New Jersey Department of Transportation
NOAA	National Oceanic and Atmospheric Administration
NRDA	Natural Resource Damage Assessment
NS&T	National Status and Trends
OBS	Optical Backscatter Sensor
OMR	Office of Maritime Research
OPR	Ongoing Precision and Recovery
OSHA	Occupational Safety and Health Administration
OU	Operating Unit
PA	Performance Audit
PAH	Polycyclic Aromatic Hydrocarbon
PAR	Pathways Analysis Report
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
Pb-210	Lead-210
PCB	Polychlorinated Biphenyl
PCDD	Polychlorinated Dibenzodioxin
PCDF	Polychlorinated Dibenzofuran
PE	Performance Evaluation
pg/g	picogram/gram
pg/L	picogram per Liter
PID	Photoionization Detector
PM	Project Manager
Po-210	Polonium-210
POC	Particulate Organic Carbon
ppb	parts per billion
ppm	parts per million
ppq	parts per quadrillion
ppt	parts per trillion
PREmis	Passaic River Estuary Management Information System
PRG	Preliminary Remediation Goals
PSO	Project Safety Officer
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan

QC	Quality Control
QCCS	Quality Control Check Sample
QCP	Quality Control Plan
QCS	Quality Control Standard
QCT	Quality Control Team
QL	Quantitation Limit
R	Recovery
RA	Risk Assessment
RBC	Risk Based Concentration
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study
RL	Reporting Limit
RM	River Mile
ROC	Receptors of Concern
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center
SAV	Submerged Aquatic Vegetation
SDG	Sample Delivery Group
SEM	Simultaneously Extractable Metals
SMO	Sample Management Officer
SOP	Standard Operating Procedure
SOW	Statement of Work
SPI	Sediment Profile Imagery
SPMD	Semi-Permeable Membrane Device
SQO	Site Quality Control Officer
SSS	Side Scan Sonar
STL	Severn Trent Laboratories
SVOC	Semi-Volatile Organic Compound
TAC	Technical Advisory Committee
TAL	Target Analyte List
TAT	Turnaround Time
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TD	Technical Director
TDS	Total Dissolved Solids
TEQ	Toxic Equivalency Quotient
TIC	Tentatively Identified Compound

Th-234	Thorium-234
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbons
TSA	Technical System Audit
TSI	Tierra Solutions, Inc.
TSS	Total Suspended Solids
µg/kg	microgram per kilogram
µm	micron or micrometer
USACE-KC	United States Army Corps of Engineers-Kansas City District
USACE-NY	United States Army Corps of Engineers-New York District
USCG	United States Coast Guard
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service
USGS	United States Geological Survey
VOC	Volatile Organic Compound
VSS	Volatile Suspended Solids
WP	Work Plan
WRDA	Water Resources Development Act
WWTP	Wastewater Treatment Plant

TABLE 1-1
Technical Advisory Committee Members

Name	Affiliation
Richard Bopp, PhD	Rensselaer Polytechnic Institute
Bruce Brownawell, PhD	State University of New York at Stony Brook
Jon Butcher, PhD, PH	Tetra Tech, Inc.
Frank Gobas, PhD	Simon Fraser University
John Henningson, PE	Henningson Environmental Services
Willy Lick, PhD	University of California at Santa Barbara
Richard Luthy, PhD, PE	Stanford University
Rob Mason, PhD	University of Maryland

TABLE 2-1
Reporting Limits for TAL Metals plus Cyanide
(Requested through USEPA-CLP)

Inorganic Parameter	Water (µg/L)[d]	Sediment dry weight (mg/kg)[c,d]
Aluminum	200	20
Antimony[a]	2	1
Arsenic[a]	0.5	0.25
Barium[a]	10	5
Beryllium[a]	1	0.25
Cadmium[a]	1	0.25
Calcium	5000	500
Chromium[a]	2	1
Cobalt[a]	1	0.5
Copper[a]	1	1
Iron	100	10
Lead[a]	1	0.5
Magnesium	5000	500
Manganese[a]	1	0.5
Mercury[a]	0.05	0.03
Nickel[a]	1	0.5
Potassium	5000	500
Selenium[a]	1	0.5
Silver[a]	0.5	0.25
Sodium	5000	500
Thallium[a]	1	0.5
Titanium[b]	10	100
Vanadium[a]	1	0.5
Zinc[a]	2	1
Cyanide[a]	5	2.5

Notes:

a. Identified as a COPC/COPEC in the PAR.

b. Identified as a COPC/COPEC, but not on the standard CLP list, so will be requested for analysis under the CLP flex clause.

c. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

d. The target RLs have been reviewed with the USEPA CLP Region 2 Program Officer and will be requested under the CLP flex clause to meet the project data needs.

TABLE 2-2
Reporting Limits for TCL VOCs
(Requested through EPA-CLP)

VOCs	Water (µg/L)[c]	Sediment dry weight (µg/kg)[b,c]
Dichorodifluoromethane	0.5	5
Chloromethane	0.5	5
Vinyl Chloride	0.5	5
Bromomethane	0.5	5
Chloroethane	0.5	5
Trichlorofluoromethane	0.5	5
1,1-Dichloroethene	0.5	5
1,1,2-Trichloro-1,2,2-trifluoroethane	0.5	5
Acetone	5	10
Carbon Disulfide	0.5	5
Methyl Acetate	0.5	5
Methylene Chloride[a]	0.5	5
Trans-1,2-Dichloroethene[a]	0.5	5
Methyl tert-Butyl Ether	0.5	5
1,1-Dichloroethane	0.5	5
cis-1,2-Dichloroethene[a]	0.5	5
2-Butanone[a]	5	10
Bromochloromethane	0.5	5
Chloroform	0.5	5
1,1,1-Trichloroethane	0.5	5
Cyclohexane	0.5	5
Carbon Tetrachloride	0.5	5
Benzene[a]	0.5	5
1,2-Dichloroethane	0.5	5
1,4-Dioxane	20	100
Trichloroethene	0.5	5
Methlycyclohexane	0.5	5

Notes:

a. Identified as a COPC/COPEC in the PAR.

b. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

c. The target RLs have been reviewed with the USEPA CLP Region 2 Program Officer.

TABLE 2-2 (Continued)
Reporting Limits for TCL VOCs
(Requested through USEPA-CLP)

VOCs	Water (µg/L)[c]	Sediment dry weight (µg/kg)[b,c]
1,2-Dichloropropane	0.5	5
Bromodichloromethane	0.5	5
cis- 1,3-Dichloropropene	0.5	5
4-Methyl-2-pentanone	5	10
Toluene	0.5	5
Trans-1,3-Dichloropropene	0.5	5
1,1,2-Trichloroethane	0.5	5
Tetrachloroethene	0.5	5
2-Hexanone	0.5	5
Dibromochloromethane	10	5
1,2-Dibromoethane	0.5	5
Chlorobenzene[a]	0.5	5
Ethylbenzene[a]	0.5	5
o-Xylene	0.5	5
M, p-Xylene	0.5	5
Styrene	0.5	5
Bromoform	0.5	5
Isopropylbenzene	0.5	5
1,1,2,2-Tetrachloroethane	0.5	5
1,3-Dichlorobenzene	0.5	5
1,4-Dichlorobenzene[a]	0.5	5
1,2-Dichlorobenzene	0.5	5
1,2-Dibromo-3-chloropropane	0.5	5
1,2,4-Trichlorobenzene[a]	0.5	5
1,2,3-Trichlorobenzene	0.5	5

Notes:

a. Identified as a COPC/COPEC in the PAR.

b. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

c. The target RLs have been reviewed with the USEPA CLP Region 2 Program Officer.

TABLE 2-3
Reporting Limits for TCL SVOCs including PAHs
(Requested through USEPA-CLP)

SVOCs (including PAHs)	Water (µg/L)[c]	Sediment dry weight (µg/Kg)[c,d]
Benzaldehyde	5.0	170
Phenol	5.0	170
bis-(2-Chloroethyl)ether	5.0	170
2-Chlorophenol	5.0	170
2-Methylphenol	5.0	170
2,2'-Oxybis (1-Chloropropane)	5.0	170
Acetophenone	5.0	170
4-Methylphenol	5.0	170
N-Nitroso-di-n-propylamine	5.0	170
Hexachloroethane	5.0	170
Nitrobenzene	5.0	170
Isophorone	5.0	170
2-Nitrophenol	5.0	170
2,4-Dimethylphenol	5.0	170
bis-(2-Chloroethoxy)methane	5.0	170
2,4-Dichlorophenol	5.0	170
Naphthalene[a]	0.1	3.3
4-Chloroaniline	5.0	170
Hexachlorobutadiene	5.0	170
Caprolactam	5.0	170
4-Chloro-3-methylphenol	5.0	170
1-Methylnaphthalene	5.0	170
2-Methylnaphthalene[a]	0.1	3.3
Hexchlorocyclo-pentadiene	5.0	170
2,4,6-Trichlorophenol	5.0	170
2,4,5-Trichlorophenol	5.0	170
1,1'-Biphenyl[a]	0.1	3.3

Notes:

a. Identified as a COPC/COPEC in the PAR.

b. Identified as a COPC/COPEC, but not on the standard CLP list, will be requested for analysis under the CLP flex clause.

c. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

d. The target RLs have been reviewed with the USEPA CLP Region 2 Program Officer and will be requested under the CLP flex clause to meet the project data needs.

TABLE 2-3 (Continued)
Reporting Limits for TCL SVOCs including PAHs
(Requested through USEPA-CLP)

SVOCs (including PAHs)	Water (µg/L)[c]	Sediment dry weight (µg/Kg)[c,d]
2-Chloronaphthalene	5.0	170
2-Nitroaniline	5.0	170
Dimethylphthalate	5.0	170
2,6-Dinitrotoluene	5.0	170
Acenaphthylene[a]	0.1	3.3
3-Nitroaniline	10	330
Acenaphthene[a]	0.1	3.3
2,4-Dinitrophenol	5.0	170
4-Nitrophenol	5.0	170
Dibenzofuran	5.0	170
2,4-Dinitrotoluene	5.0	170
Diethylphthalate	5.0	170
Fluorene[a]	0.1	3.3
4-Chlorophenyl-phenyl ether	5.0	170
4-Nitroaniline	10	330
4,6-Dinitro-2-methylphenol	10	330
N-Nitrosodiphenylamine[a]	0.1	3.3
1,2,4,5-Tetrachlorobenzene	5.0	170
4-Bromophenyl-phenylether	5.0	170
Hexachlorobenzene	0.5	17
Atrazine	5.0	170
Pentachlorophenol	10	330
Phenanthrene[a]	0.1	3.3
Anthracene[a]	0.1	3.3
Carbazole[a]	0.1	3.3

Notes:

a. Identified as a COPC/COPEC in the PAR.

b. Identified as a COPC/COPEC, but not on the standard CLP list, will be requested for analysis under the CLP flex clause.

c. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

d. The target RLs have been reviewed with the USEPA CLP Region 2 Program Officer and will be requested under the CLP flex clause to meet the project data needs.

TABLE 2-3 (Continued)
Reporting Limits for TCL SVOCs including PAHs
(Requested through USEPA-CLP)

SVOCs (including PAHs)	Water (µg/L)[c]	Sediment dry weight (µg/Kg)[c,d]
Di-n-butylphthalate	5.0	170
Fluoranthene[a]	0.1	3.3
Pyrene[a]	0.1	3.3
Butylbenzylphthalate[a]	0.1	3.3
3,3',-Dichlorobenzidine	5.0	170
Benzo(a)anthracene[a]	0.1	3.3
Chrysene[a]	0.1	3.3
bis(2-Ethylhexyl)phthalate[a]	0.1	3.3
Di-n-octylphthalate[a]	0.1	3.3
Benzo(b)fluoranthene[a]	0.1	3.3
Benzo(k)fluoranthene[a]	0.1	3.3
Benzo(a)pyrene[a]	0.1	3.3
Indeno(1,2,3-cd)-pyrene[a]	0.1	3.3
Dibenzo(a,h)-anthracene[a]	0.1	3.3
Benzo(g,h,i)perylene[a]	0.1	3.3
2,3,4,6-Tetrachlorophenol	5.0	170
Benzo(e)pyrene[b]	0.1	3.3
1-Methyl-phenanthrene[b]	0.1	3.3
2,3,5-Trimethylnaphthalene[b]	0.1	3.3
2,6-Dimethylnaphthalene[b]	0.1	3.3
Perylene[b]	0.1	3.3
Dibenzothiophene[b]	0.1	3.3

Notes:

a. Identified as a COPC/COPEC in the PAR.

b. Identified as a COPC/COPEC, but not on the standard CLP list, so will be requested for analysis under the CLP flex clause.

c. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

d. The target RLs have been reviewed with the USEPA CLP Region 2 Program Officer and will be requested under the CLP flex clause to meet the project data needs.

TABLE 2-4
Reporting Limits for PCB Aroclors
(Requested through USEPA-CLP)

PCB-Aroclors[a]	Water (µg/L)[c]	Sediment Dry weight (µg/Kg)[b,c]
Aroclor 1016	0.1	3.3
Aroclor 1221	0.1	3.3
Aroclor 1232	0.1	3.3
Aroclor 1242	0.1	3.3
Aroclor 1248	0.1	3.3
Aroclor 1254	0.1	3.3
Aroclor 1260	0.1	3.3
Aroclor 1262	0.1	3.3
Aroclor 1268	0.1	3.3

Notes:

a. PCBs were identified as COPCs/COPECs in the PAR.

b. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

c. The target RLs have been reviewed with the USEPA CLP Region 2 Program Officer and will be requested under the CLP flex clause to meet the project data needs.

TABLE 2-5
Reporting Limits for Chlorinated Biphenyls by USEPA Method 1668, Revision A by HRGC/HRMS

Parameters[a]	Water (pg/L)[b,c,e]	Sediment/Solids/Non-Aqueous samples dry weight (pg/g)[b,c,d,e]
All the individual congeners PCB-1 through PCB-209[f,g]	2.0 -20	0.2-2.0

Notes:

a. PCBs were identified as COPCs/COPECs in the PAR.

b. The above target reporting limit goals are based upon detection limits on 1 liter aqueous samples or 10 gram solid samples, but reporting limits will be proportionately lower for larger volume samples.

c. The specific detection limits are highly matrix dependent.

d. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

e. The lab will be required to report congener and sample specific detection limits which may be lower.

f. Method 1668A can detect all 209 congeners, but only 125 to 150 can be completely resolved. Co-eluting congeners may vary among laboratories.

g. The PCB toxicity equivalent (PCBTEQ) and the PCB homologue distribution are calculated from the concentrations of the individual congeners.

TABLE 2-6
Reporting Limits for Dioxins/Furans
by USEPA Method 1613 Tetra through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS

Parameter[a]	Water (pg/L)[b,c]	Sediment dry weight (ng/kg)[b,c,d]
2,3,7,8-TCDD	5	0.5
1,2,3,7,8-PeCDD	25	2.5
1,2,3,4,7,8-HxCDD	25	2.5
1,2,3,6,7,8-HxCDD	25	2.5
1,2,3,7,8,9-HxCDD	25	2.5
1,2,3,4,6,7,8-HPCDD	25	2.5
OCDD	50	5.0
2,3,7,8-TCDF	5	0.5
1,2,3,7,8-PECDF	25	2.5
2,3,4,7,8-PECDF	25	2.5
1,2,3,4,7,8-HXCDF	25	2.5
1,2,3,6,7,8-HXCDF	25	2.5
2,3,4,6,7,8-HXCDF	25	2.5
1,2,3,7,8,9-HXCDF	25	2.5
1,2,3,4,6,7,8-HPCDF	25	2.5
1,2,3,4,7,8,9-HPCDF	25	2.5
OCDF	50	5.0

Notes:

a. Dioxins and furans were identified as COPCs/COPECs in the PAR.

b. The above target reporting limits goals are based upon detection limits on 1 liter aqueous samples or 10 gram solid samples, but reporting limits will be proportionately lower for larger volume samples.

c. The specific detection limits are highly matrix dependent.

d. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

TABLE 2-7
Reporting Limits for Dioxin and PCB Screening (Immunoassay) by a modified version of EPA SW-846-4025, Screening for Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by Immunoassay.

Parameter	Sediment dry weight (pg/g)[a,b]
Total Dioxin/Furan TEQ ($TEQ_{D/F}$)	Approx. 20
Total Coplanar PCB TEQ (TEQ_{PCB})	Approx. 20

Notes:

a. The specific detection limits are highly matrix dependent.

b. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

TABLE 2-8

Reporting Limits for PAHs Analyzed by Non-CLP Laboratory

Modified USEPA Method 8270 - GC-MS-SIMs

Parameter[a]	Water (µg/L)[b,c]	Solids on Filter, Dry Sediment (µg/kg)[b,c,d]
1-Methylphenanthrene	0.1	3.3
2,3,5-Trimethylnaphthalene	0.1	3.3
2,6-Dimethylnaphthalene	0.1	3.3
2-Methylnaphthalene	0.1	3.3
Acenaphthene	0.1	3.3
Acenaphthylene	0.1	3.3
Anthracene	0.1	3.3
Fluorene	0.1	3.3
Naphthalene	0.1	3.3
Phenanthrene	0.1	3.3
Benzo[a]pyrene	0.1	3.3
Benzo[b]fluoranthene	0.1	3.3
Benzo[e]pyrene	0.1	3.3
Benzo[g,h,i]perylene	0.1	3.3
Benzo[k]fluoranthene	0.1	3.3
Benzo(a)anthracene	0.1	3.3
Chrysene	0.1	3.3
Dibenzo[a,h]anthracene	0.1	3.3
Fluoranthene	0.1	3.3
Indeno[1,2,3-c,d]-pyrene	0.1	3.3
Perylene	0.1	3.3
Pyrene	0.1	3.3
Dibenzothiophene	0.1	3.3

Notes:

a. These PAHs were identified as COPCs/COPECs in the PAR.

b. The above target reporting limits goals are based upon detection limits on 1 liter aqueous samples or 10 gram solid samples, but reporting limits will be proportionately lower for larger volume samples.

c. The specific detection limits are highly matrix dependent.

d. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

TABLE 2-9
Reporting Limits for Pesticides
By Modified USEPA Method SW-846 8081

Parameter	Water (µg/L)[b,c]	Sediment dry weight (µg/Kg)[b,c,d]
alpha-BHC[a]	0.005	0.2
beta-BHC[a]	0.005	0.2
delta-BHC	0.005	0.2
gamma-BHC (Lindane)[a]	0.005	0.2
Hetachlor[a]	0.005	0.2
Aldrin[a]	0.005	0.2
Heptachlor epoxide[a]	0.005	0.2
Endosulfan I[a]	0.005	0.2
Dieldrin[a]	0.005	0.2
4,4'-DDE[a]	0.005	0.2
Endrin[a]	0.005	0.2
Endosufan II[a]	0.005	0.2
4,4'-DDD[a]	0.005	0.2
Endosulfan sulfate	0.005	0.2
4,4'-DDT[a]	0.005	0.2
Methoxychlor[a]	0.01	0.3
Endrin ketone	0.005	0.2
Endrin aldehyde	0.005	0.2
alpha-Chlordane[a]	0.005	0.2
gamma-Chlordane[a]	0.005	0.2
Toxaphene[a]	0.5	17
2,4'-DDD[a]	0.005	0.2
2,4'-DDE[a]	0.005	0.2
2,4'-DDT[a]	0.005	0.2

Notes:

a. Identified as a COPC/COPEC in the PAR.

b. The above target reporting limits goals are based upon detection limits on 1 liter aqueous samples or 10 gram solid samples, but reporting limits will be proportionately lower for larger volume samples.

c. The specific detection limits are highly matrix dependent.

d. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

TABLE 2-10
Reporting Limits for Chlorinated Herbicides
by USEPA SW-846 Method 8151A, Chlorinated Herbicides by GC

Parameter[a]	Water (µg/L)[b]	Sediment/Solid (µg/kg)[b,c]
2,4-D	2	140
2,4-DB	2	160
2,4,5-TP (Silvex)	1	20
2,4,5-T	1	20

Notes:

a. Identified as COPCs/COPECs in the PAR.

b. The specific detection limits are highly matrix dependent.

c. The RLs listed above for sediment/solids are based upon the whole sample analysis. The lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may be higher.

TABLE 2-11
Reporting Limits for Butyl tin Compounds

Parameter[a]	Sediment/Solid (µg/kg)[b,c,d]
Monobuyltin	1.0
Dibutyl tin	1.3
Tributyl tin	1.5
Tetrabutyl tin	1.7

Notes:

a. Identified as COPCs/COPECs in the PAR.

b. The specific detection limits are highly matrix dependent.

c. The RLs listed above are based upon the whole sample analysis. The lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may be higher.

d. Lab will analyze using a lab-specific GC method which is targeted to achieve the RLs above.

TABLE 2-12
Reporting Limits for TPH
by the New Jersey DEP Method Using GC/FID

Parameter[a]	Dry Sediment (mg/kg)[b,c,d]
TPH	20

Notes:

a. Identified as a COPC/COPEC in the PAR.

b. The specific detection limits are highly matrix dependent.

c. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

d. The scope of method NJDEP OQA-QAM-025-10/91, Quantitation of Semi-Volatile Petroleum Products in Water, Soil, Sediment, and Sludge, is applicable to "quantitative analysis of environmental samples (water, soil, sediment, and sludge) for residues from commercial petroleum products such as crude oil, diesel fuel, waste oil, fuel oils Nos. 2-6, lubricating oil, processed oils, and bunker fuel ... The gas chromatographic conditions are not designed for compounds with carbon numbers greater than C40."

TABLE 2-13
Reporting Limits for TOC, DOC, and POC

TOC and DOC
by EPA SW-846-9060 – Total Organic Carbon and Determination of Total Organic Carbon in Sediment (7-27-88, L. Kahn, USEPA).

Parameter	Whole Water (mg/L)	Filtered Water (mg/L)[a]	Sediment (mg/kg)
Total Organic Carbon	1	n/a	100
Dissolved Organic Carbon	n/a	1	n/a

Note:

a. To measure DOC a portion of the aqueous sample must be first filtered through a 0.45 µm filter.

POC
by USEPA Method 440.0 Particulate Organic Carbon or modified Lloyd Kahn

Parameter	Water (µg carbon /L)[a]
Particulate Organic Carbon	65

Note:

a. Refer to Section 12.0 in Method 440.0 for the equations to calculate POC concentration in a sample.

TABLE 2-14
Reporting Limits for Trace Metals

Total and Dissolved Mercury
by Method 1631 and Methyl Mercury by USEPA Method 1630

Parameter[a]	Water (Fresh and Brackish) (ng/L)[b]	Sediment dry weight (ng/g)[b,c]
Total Mercury	0.3	0.4
Methyl Mercury	0.06	0.2

Notes:

a. Mercury has been identified as a COPC/COPEC in the PAR.

b. The specific detection limits are highly matrix dependent.

c. The lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

Arsenic Speciation
by USEPA Method 1632

Parameter[a]	Dry Sediment (mg/kg)[b,c]
Arsenic (total and dissolved)	0.2
Arsenic (II) (total and dissolved)	0.2
Arsenic (V) (total and dissolved)	0.2

Notes:

a. Arsenic has been identified as a COPC/COPEC in the PAR.

b. The specific detection limits are highly matrix dependent.

c. The lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

Hexavalent Chromium
By EPA Method SW-846-7199 by Ion Chromatography

Parameter[a]	Aqueous (µg/L)[b]	Sediment dry weight (µg/kg)[b,c]
Chromium VI	1	10

Notes:

a. Chromium has been identified as a COPC/COPEC in the PAR.

b. The specific detection limits are highly matrix dependent.

c. The lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

TABLE 2-15
Reporting Limits for
AVS and SEM in Sediment

Parameter	Sediment dry weight (µmoles/g)	Sediment Extract (µmoles/g)
Acid Volatile Sulfide (AVS)	1.0	n/a
SEM-cadmium, mg/kg	n/a	1
SEM-copper, mg/kg	n/a	1
SEM-lead, mg/kg	n/a	0.5
SEM-mercury, mg/kg	n/a	0.02
SEM-nickel, mg/kg	n/a	0.5
SEM-zinc, mg/kg	n/a	1

TABLE 2-16
Reporting Limits for Wet Chemistry

Phosphate and Orthophosphate by EPA Method 365.2(Colorimetric, Ascorbic Acid Method)

Parameter	Water (mg/L)
Phosphate (P)	0.01
Total Orthophosphate (P, ortho)	0.01

Nitrogen (Kjeldahl) by EPA Method 351.3

Parameter	Water (mg/L)	Sediment dry weight (mg/kg)
Nitrogen (Total Kjeldahl)	1	150

Ammonia by EPA Method 350.2

Parameter	Water (mg/L)
Ammonia as N	0.02

Reporting Limits for Chlorophyll a by SM 10200-H

Parameter	Water (mg/m^3)
Chlorophyll a (Chl a)	1.0

TABLE 2-17
Reporting Limits for COD and BOD

COD by USEPA Method 410.4

Parameter	Water (mg/L)
Chemical Oxygen Demand	20

BOD_5 by USEPA Method 405.1

Parameter	Water (mg/L)
Biochemical Oxygen Demand	2.0

TABLE 2-18
Reporting Limits for TDS, TSS, VSS, and pH

TDS by USEPA Method 160.1 Method for Chemical Analysis of Water and Wastes, EPA600/4/79/020

Parameter	Water (mg/L)
Total Dissolved Solids	1

Suspended Sediment by USEPA Method 160.2 Method for Chemical Analysis of Water and Wastes, EPA600/4/79/020

Parameter	Water (mg/L)[a]
Suspended Sediment	1

Note:

a. The RL is based on the analysis of the entire 1-L sample, not a 100-mL aliquot, as specified in the method.

VSS by USEPA Method 160.4 Method for Chemical Analysis of Water and Wastes, EPA600/4/79/020

Parameter	Water (mg/L)
Volatile Suspended Solids	1

Corrosivity (pH) by USEPA SW-846- 9045C

Parameter	Water	Sediment
Corrosivity (pH)	All ranges	All ranges

TABLE 2-19
Reporting Limits for Radionuclides

Parameter	Sediments (pCi/g)[a]
Cesium-137	< 0.05 pCi/g
Beryllium-7	< 0.3 pCi/g
Lead -210	< 0.1 pCi/g

Notes:

a. Lab will report dry weight results; dependent upon the sample moisture content and matrix effects the RLs achieved may in some cases be higher.

TABLE 2-20
Reporting Limits for Cation Exchange Capacity
by SW-846, Method 9081, Cation-Exchange Capacity of Soils (Sodium Acetate)

Parameter	Sediment
Cation Exchange Capacity	Follow the reporting requirements of the method

TABLE 2-21
Reporting Limits for Geotechnical Parameters

Percent (%) Moisture by ASTM D 2974 -Test Method A

Parameter	Sediment (%)
% Moisture content, total mass	0.1
% Moisture, oven-dried mass	0.1

Grain Size by ASTM Method D422 or D4464

Parameter	Sediment
Grain Size Distribution	Follow the reporting requirements in D422 (sieve and hydrometer) or D4464 Laser Light Scattering.

Specific Gravity (Density) by ASTM D854

Parameter	Sediment
Specific Gravity	Follow the reporting requirements in D854.

Atterberg Limits by ASTM D4318

Parameter	Sediment
Atterberg Limits	Follow the reporting requirements in D4318.

TABLE 3-1

Sample Bottle, Volume, Preservation, and Holding Times for Analysis of Non-Organics in Sediments

Parameter Analyzed	Approximate Sample Size	Container Material	Preservation	Holding Time
TAL and SEM Metals	16 oz.	G, P	4°C	6 months
Cyanide				14 days
Mercury				28 days
Arsenic Speciation	4 oz.	G, P	No head space, kept field moist, store at 4°C, do not allow to air dry.	28 days at 4°C (Frozen samples can be stored for up to one year)
Trace Mercury by EPA 1631	4 oz.[a]	G, P	Frozen upon collection and shipped frozen.	Stored frozen for up to 1 year
Methyl Mercury By EPA 1630	4 oz.	G, P	Frozen upon collection and shipped frozen.	Stored frozen for up to 1 year
Chromium, Hexavalent	8 oz	G, P	No head space, kept field moisture, store at 4°C, do not allow to air dry.	24 hours
AVS-SEM	8 oz.	G, P	No head space, kept field moist, store at 4°C; do not allow to air dry.	14 days
Nitrogen Kjeldahl	4 oz	G, P	Cool 4°C	28 days
Radionuclides	16 oz.	G, P	None	1 month[b]

G=Glass
P = Plastic

a. The laboratory will supply the sample bottles for trace metals analyses

b. Shortest radionuclide holding time listed (1 month for Be-7).

TABLE 3-2

Sample Bottle, Volume, Preservation, and Holding Times for Analysis of Organics in Sediments

Parameter Analyzed	Approximate Sample Size	Container Material	Preservation	Holding Time
VOC	3 x 5g EnCore™	EnCore™	4°C	48 hrs. to extraction; 8 days to analysis
SVOC	2-8 oz.	G	4°C	7 days to extraction, 40 days until analysis
Aroclor PCBs				
Pesticides	4 oz	G	4°C	7 days to extraction, 40 days until analysis
PCB Congeners 1668A	8 oz.	G, Amber	Maintain in dark at <4°C from time of collection until receipt at lab	If stored at < -10°C solid, multiphase samples can be stored for up to one year. Sample extracts can be stored at < -10 °C for up to one year.
Dioxins/furans 1613	8 oz.	G, Amber		
Dioxin TEQ/ PCB TEQ Immunoassay Screening	16 oz	G, Amber		
Chlorinated Herbicides	8 oz.	G, Amber	4°C in dark	7 days to extraction, 40 days until analysis
non-CLP PAHs	4 oz.	G	4°C	7 days to extraction, 40 days until analysis
TOC	4 oz.	G	4°C	28 days
Butyl tins	4 oz.	G	4°C	14 days to extraction, 40 days until analysis
TPH	4 oz.	G	4°C	14 days to extraction, 40 days until analysis

G = Glass

TABLE 3-3

Sample Bottle, Volume, Preservation, and Holding Times for Analysis of Geotechnical Parameters in Sediment

Parameter Analyzed	Approximate Volume	Container Material	Preservation	Holding Time
CEC	8 oz.	G, P	4°C	6 months
% Moisture	4 oz.	G, P	Airtight container cooled to 4°C	Test as soon as practical after sampling
Engineering Parameters: Grain size (D422) Density (Specific Gravity) Atterberg Limits	64 oz. (high resolution cores will only be analyzed for grain size; one core tube is required)	G, P	Airtight container	6 months (Grain size and Atterberg Limits should be tested as soon as practical)
Grain Size by ASTM D4464 Laser Light Scattering	4 oz	G, P	Airtight container	6 months (Grain size should be tested as soon as practical)

G=Glass
P = Plastic

TABLE 3-4
Sample Bottle, Volume, Preservation, and Holding Times for Analysis of Organics in Water

Parameter Analyzed	Approximate Volume	Container Material	Preservation	Holding Time
VOC	3 - 40 mL VOC vials	G, Teflon-lined septa	4°C; no bubbles or headspace, HCl to pH<2	14 days
SVOC	2-1 liter	G, Amber	4°C	7 days to extraction, 40 days until analysis
Pesticides[a]	2-1 liter	G, Amber	4°C	
Aroclor PCBs	2-1 liter	G, Amber	4°C	
PCB Congeners 1668A[a]	2-1 liter	G, Amber	Maintain in dark at 0-4°C from time of collection until receipt at lab	At 0-4°C in the dark can be stored for up to one year. Extracts can be stored at < -10 °C for up to one year.
Dioxins/furans 1613B[a]	2-1 liter	G, Amber	Maintain in dark at 0-4°C from time of collection until receipt at lab	At 0-4°C in the dark can be stored for up to one year. Extracts can be stored at < -10 °C for up to one year.
Chlorinated Herbicides	2-1 liter	G, Amber	4°C in the dark	7 days to extraction, 40 days until analysis
TOC	250 mL	G	4°C; H_2SO_4 to pH<2	28 days
DOC	250 mL	G	4°C; Filter within 48 hours than H_2SO_4 to pH<2	28 days
POC	1 liter	P or G	Store at 4°C until filtration	Must be filtered within 7 days and filters analyzed within 7 days.

G = Glass
P = Plastic

a. Large volume aqueous samples will be collected for organic parameters in water including PCB congeners, Dioxins/Furans, and Pesticides.

TABLE 3-5
Sample Bottle, Volume, Preservation, and Holding Times for Analysis of Inorganics in Water

Parameter Analyzed	Approximate Volume	Container Material	Preservation	Holding Time
TAL Metals plus Titanium	1 liter[a]	G, P	HNO_3 to pH <2, Cool at 4°C	6 months
Cyanide	1 liter	G, P	Ascorbic acid NaOH to pH >12 . Cool at 4°C	14 days
Trace Mercury[b]	1 liter[a]	G, P	HNO_3 to pH <2, Cool at 4°C	28 days
Methyl Mercury[b, c]	0.5 liter	G, P	Acidify; Cool at 4°C	6 months
Chromium, Hexavalent	0.5 liter	G, P	Cool at 4°C	24 hours[d]

G=Glass
P = Plastic

a. For metals, whenever samples are sent for total and dissolved analyses, 2 1-liter bottles must be collected.

b. The analytical laboratory will supply the bottles for trace metals analyses.

c. Saline samples must be preserved with 2 mL/L of 9 M H_2SO_4 solution. Fresh water samples are preserved with 4 mL/L of concentrated HCl. Aqueous samples must be acid preserved within 48 hours of collection. Acid preserved samples are stable for at least six months, if kept dark and cool.

d. For this project if the sample is analyzed on the next calendar day after collection, it will be considered that it has met the holding time.

TABLE 3-6
Sample Bottle, Volume, Preservation, and Holding Times for Analysis of Wet Chemistry Parameters in Water

Parameter analyzed	Approximate Volume	Container Material	Preservation	Holding Time
Total Phosphate & Orthophosphate	500 mL	G, P	H_2SO_4 to pH <2 Cool 4°C	28 days
Nitrogen (Kjeldahl)	500 mL	G, P	H_2SO_4 to pH <2 Cool 4°C	28 days
Ammonia	1 Liter	G, P	H_2SO_4 to pH <2. Cool at 4°C (no headspace)	28 days
COD	250 mL	G, Amber	H_2SO_4 to pH <2 Cool 4°C	28 days
BOD	1 liter	G, P	Cool 4°C, store in dark	48 hours
TDS				
TSS				
VSS	1 Liter[a]	G, P	Cool 4°C	7 days
Chlorophyll a	4 Liters	G, P	Filter in subdued light as soon as possible. Freeze filters. If storage of water is necessary store at 4°C.	48 hours to filtration. Frozen filters can be held 3 weeks.
pH	b	G	Cool 4°C	24 hours

G=Glass
P = Plastic

a. Whenever TDS, TSS, and VSS are analyzed for a sample, the analyses will be conducted on one 1-L aliquot. First, the entire 1-L sample will be filtered through a 0.45 µm filter. TDS will be determined on the water that passes through the filter; TSS will be determined from the weight of the solids retained on the filter; and VSS will be determined by igniting the filter.

b. For this project pH will be measured using a field instrument or on occasion by the lab on a portion of sample collected for another test.

TABLE 4-1
Analytical Methods for Inorganic Parameters

Parameter	Technique	Water	Sediment
TAL Metals plus titanium	ICP-AES,ICP-MS	EPA-CLP (ILM0.5.3) with flex clause	
Cyanide	Colorimetric	EPA-CLP (ILM0.5.3)	
Total Mercury	CVAFS	EPA-CLP (ILM0.5.3) with flex clause	
Arsenic, Arsenic III and Arsenic V	Hydride Generation Quartz Furnace Atomic Absorption	NA	EPA 1632A plus modifications for extraction of sediment
Trace Mercury	Purge and Trap plus CVAFS	EPA 1631	EPA 1631 plus modifications for extraction of sediment
Methyl Mercury	CVAFS	EPA 1630	EPA 1630 plus modifications for extraction of sediment
Chromium, Hexavalent	Ion Chromatography	7199[a]	7199/3060A[a]
Acid Volatile Sulfide	Acidification to H_2S than purge and trap	NA	EPA 821-R-91-100[b]
SEM Metals: Cd, Pb, Hg, Ni and Zn	ICP-AES or ICP-MS or GFAA and CVAA.	NA	SW-846 methods[a] or other approved USEPA methods for metals

Note: Samples for trace metal analyses will be collected using procedures based upon EPA Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria.

a. USEPA SW-846 "Test Methods for Evaluating Solid Waste," Third Edition, December 1996 including promulgated final update III.

b. USEPA 821-R-91-100, Draft Analytical Method for the Determination of Acid Volatile Sulfide and Selected Simultaneously Extractable Metals in Sediment, December 1991.

c. Method 1632 Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry, Revision A, August 1998.

TABLE 4-2
Analytical Methods for Organic Parameters

Parameter	Technique	Water	Sediment
VOC	GC-MS	EPA-CLP SOM1.0	
SVOCs including PAHs and PCB Aroclors	GC-MS-SIM GC-MS GC-ECD	EPA-CLP SOM1.0, with flex cause options to achieve requested RLs	
PAHs	GC-MS-SIM	8270 [a] (modified)	
Pesticides	GC-MS & GC-ECD	8081 [a] (modified)	
PCB congeners	HRGC-HRMS	EPA 1668A[b]	
Dioxins/furans	HRGC-HRMS	EPA 1613B[c]	
Screening for $Dioxin_{TEQ}$ and PCB_{TEQ}	Extraction plus Immunoassay	NA	4025[a] (modified[d])
Chlorinated Herbicides	GC	8151A[a]	8151A[a]
Butyl tins	GC	NA	Lab specific SOP
TOC	Combustion	NA	Lloyd Kahn[e]
POC	Elemental Analyzer	EPA 440.0 or Lloyd Kahn[e]	NA
TOC and DOC[f]	Carbonaceous Analyzer	9060[f]	NA
TPH	GC	NA	NJDEP

a. USEPA SW-846 "Test Methods for Evaluating Solid Waste," Third Edition, December 1996 including promulgated final update III.

b. Method 1668, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/MRMS, EPA-821-R-00-002, December 1999.

c. Method 1613, Revision B: Tetra through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, October 1994.

d. Cape Technologies Technical Notes TN-004 and TN-005.

e. USEPA Region 2, Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method) July 27, 1988.

f. Determination of DOC requires that the sample be passed through a 0.45-µm filter prior to analysis to remove any particulate organic carbon. (Refer to USEPA Method 415.3, Rev. 1, June 2003 for a description of a suitable filtration procedure.)

TABLE 4-3
Analytical Methods for Wet Chemistry Parameters

Parameter	Technique	Water	Sediment
Total Phosphate and Orthophosphate	Colorimetric	EPA 365.2	NA
Nitrogen (Kjeldahl)	Distillation	EPA 351.3	
Ammonia	Colorimetric	EPA 350.2	NA
Chemical Oxygen Demand	Titration	EPA 410.4	NA
Biochemical Oxygen Demand	Membrane	EPA 405.1	NA
Total Dissolved Solids	Gravimetric	EPA 160.1	NA
Total Suspended Solids	Gravimetric	EPA 160.2	NA
Volatile Suspended Solids	Gravimetric	EPA 160.4	NA
Chlorophyll a	Fluorescence	SM 10200-H	NA
pH	Electrode	9045C[a]	

a. USEPA SW-846 "Test Methods for Evaluating Solid Waste," Third Edition, December 1996 I, including promulgated final update III.

TABLE 4-4
Analytical Methods for Radiochemistry Parameters

Parameter	Technique	Water	Sediment
Be-7, Cs-137	Gamma-Spec	NA	HASL-300 EML and USEPA-600[a]
Pb-210	Low Energy Gamma Spec or Alpha Spectrometry[a]	NA	

a. HASL-300 EML Procedures Manual, U.S. Department of Energy, 28th Edition, Volume 1, February 1997 and/or USEPA-600 4-80-032, Prescribed Procedures for Measurement of Radioactivity in Drinking Water, August 1980. (Cs-137 and Be-7 can be determined by Gamma Spec. Lead-210 to be determined by Low energy Gamma Spec. or HASL-300 PB-1 or Extraction Chromatography with Alpha Spectrometry 2nd decay daughter Po-210.)

TABLE 4-5
Analytical Method for Geotechnical Parameters

Parameter	Test Method
Cation Exchange Capacity	9081[a]
% Moisture	ASTM D2974, Standard Test Method for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils – Test Method A
Grain size	ASTM D422, Standard Test Method for Particle Size Analysis of Soils or ASTM D4464, Standard Method for Particle Size Distribution by Laser Light Scattering
Density (Specific Gravity)	ASTM D854, Standard Test Method for Specific Gravity of Soil Solids by Water Pyncometer
Atterberg Limits	ASTM D4318, Standard Test Method for Liquid, Plastic Limit, and Plasticity Index

a. USEPA SW-846 "Test Methods for Evaluating Solid Waste," Third Edition, December 1996 I, including promulgated final update III.

Attachment 1.1

Data Quality Objectives

Data Quality Objectives

DQOs are used to help PMs collect data of the right type, quality and quantity to support decisions. The approach to developing DQOs is an iterative one, designed to take PMs through a strategic planning process from broad project goals through a number of refining steps toward generating environmental data that will be appropriate to making the decisions needed to reach the goals.

This document begins with a "project-level" statement of the DQOs that sets the framework for addressing the environmental problems of the Study Area. The project-level DQOs focus on the information that the PM team needs to carry out an integrated CERCLA RI/FS, WRDA FS and CERCLA NRDA that will produce a comprehensive watershed plan for the Lower Passaic River.

1.0 State the Problem

The Study Area history, setting, and current conditions are summarized in Sections 1.0 through 4.0 of the Work Plan. The CSM represents the processes in the Lower Passaic River watershed that determine the transport of contaminants (Work Plan, Attachment A).

The objectives of the Lower Passaic River Restoration Project investigation activities ("the Study") are as follows:

- To characterize the nature and extent of contamination in the Lower Passaic River.
- To characterize the mechanisms governing long-term fate and transport of site contaminants.
- To assess the human health and ecological risks posed by the contamination in the Lower Passaic River.
- To characterize the function and structure of candidate restoration sites in the Lower Passaic River watershed.
- To evaluate remedial alternatives that meet both CERCLA and WRDA selection criteria to address unacceptable human health/ecological risks and provide for restoration within the Lower Passaic River watershed; as well as to evaluate options for reducing costs associated with dredging contaminated harbor sediments originating from the Passaic River.
- To support development of a NRDA under CERCLA.

2.0 Identify the Decision

To meet the objectives of the Study, the following Fundamental Questions will need to be answered during the investigation:

1. If we take no action on the River, when will the COPCs and COPECs recover to acceptable concentrations?
2. What actions can we take on the River to significantly shorten the time required to achieve acceptable or interim risk-based concentrations for human and ecological receptors?
3. Are there contaminated sediments now buried that are likely to become exposed following a major flood, possibly resulting in an increase in contaminants within the fish/crab populations?
4. What actions can we take on the River to significantly improve the functionality of the Lower Passaic River watershed?
5. If the risk assessments for Newark Bay demonstrate unacceptable risks due to contaminant export from the Passaic River, will the plan proposed to achieve acceptable risks for Passaic River receptors significantly shorten the time required to achieve acceptable or interim risk-based concentrations for receptors in Newark Bay, or will additional actions be required on the Passaic River?[1]
6. What actions can we take on the River to significantly reduce the cost of dredged material management for the navigational dredging program?
7. What actions can we take to restore injured resources and compensate the public for their lost use?

Each Fundamental Question may be divided into smaller-scale questions that are more manageable to answer through an investigation:

1, 2, 3: For the first three questions above, the following apply:

a. What are the COPCs and COPECs?
b. What is the extent and distribution of contaminants in sediment, surface water and biota? Have the sources been identified?
c. How stable are the sediments in the Study Area? Are contaminants being exported from and imported into the Study Area? How could contaminant transport be impacted by extreme events?
d. What are the quantitative human and ecological health risks posed by the contamination in the Study Area?
e. Are the human health and ecological risks unacceptable (*i.e.*, the risk range identified in the National Contingency Plan (NCP) is exceeded), and consequently, is assessment of remedial actions warranted?
f. What is the comparative performance of remedial alternatives (including potential interim remedies), based on CERCLA and WRDA criteria?
g. What are the relative risk reductions associated with the various remedial actions (including potential interim remedies) in relation to the baseline risks?

4: For the fourth question above, the following apply:

a. What are the candidate restoration sites?

[1] This question is shared with the RI/FS for the Newark Bay Study, since the actual benefits of such reduction will need to be jointly determined. A similar question to address the adequacy of any future Newark Bay plan toward achieving Passaic River goals may be included in the Newark Bay RI/FS.

b. How should candidate restoration sites be prioritized for ecosystem rehabilitation?
c. What is the type, extent and distribution of contaminants in soil, sediments, surface water and groundwater at the candidate restoration sites?
d. What is the appropriate restoration design for suitable candidate restoration sites?

5, 6: For the fifth and sixth questions above, the following apply:
a. What is the contaminant loading to Newark Bay and what is the impact of that loading on the future Newark Bay remedial action plan?
b. What is the contaminant loading to the NY/NJ Harbor and what is the impact on dredge material management for the navigational dredging program?

7: For the seventh question above, the following apply:.
a. Which of the public's natural resources are injured by the contaminants discharged by the responsible parties and how much is injured?
b. What is the pathway of the contaminants from their release to the injured resources?
c. What is the appropriate type and amount of restoration needed to restore injured resources and compensate the public for their lost use?

Clearly many of the sub-questions could be answered together and many of the sub-questions answer multiple Fundamental Questions. The iterative DQO process will continue to refine these sub-questions such that a coherent CERCLA-WRDA field sampling effort may be designed to yield appropriate environmental data.

3.0 Identify the Inputs to the Decision

The following inputs are required to answer the Fundamental Questions and refining sub-questions identified in Step 2:

I. Physical, hydraulic, hydrologic, hydrodynamic data to evaluate the stability of sediments and the degree of contaminant transport.
II. Data on biological communities to calculate human health and ecological risk, characterize injury and evaluate candidate restoration sites.
III. Chemical data in sediments, water and biota to identify COPCs/COPECs, evaluate extent of contamination, calculate human health and ecological risk and characterize injury.
IV. Exposure concentrations and ingestion rates to calculate human health and ecological risk.
V. Remedial alternative performance data to evaluate remediation and restoration options.

As discussed in Section 3.0 of the Work Plan, various tools are being developed to relate the inputs to the Fundamental Questions. Those tools include a CSM, Predictive Fate and Transport and Bioaccumulation Model and Treatability Pilot Studies.

4.0 Define the Boundaries of the Study

The physical boundaries of the Study are the watershed formed by the 17-mile tidal reach of the Passaic River and its tributaries, from Dundee Dam to Newark Bay. Since the Study Area is tidally connected to the Hackensack River, Newark Bay, Arthur Kill and Kill van Kull, the project team will need to request information from other projects underway in those water bodies in order to fully characterize Passaic River processes. Should other projects not be able to generate information needed in a timely manner, the project team may need to evaluate collecting data outside of the Lower Passaic River.

5.0 Develop a Decision Rule

The following primary decision rules will be used to answer the Fundamental Questions:

A. If the human carcinogenic risk exceeds the risk range of 1×10^{-4} to 1×10^{-6} and/or the non-carcinogenic hazard index exceeds 1, then the portions of the Study Area associated with the unacceptable human health risks will be considered for remedial action.

B. If the ecological risk hazard index exceeds 1, then the portions of the Study Area associated with the unacceptable ecological health risks will be considered for remedial action.

C. Applicable remedial alternatives will be evaluated such that they will both address unacceptable risks and be able to be integrated into planned restoration projects.

D. Candidate restoration projects will be sequenced so that any necessary remedial actions are incorporated into their implementation.

6.0 Specify Limits on Decision Errors

The general types of decision errors that may be encountered on this project are listed below along with examples of mitigative measures.

1. Laboratory Analytical Errors. It is possible that laboratory analytical data will include false negative results (low bias) or false positive results (high bias). These types of errors could lead to an underestimate of contaminated areas/inadequate remedial action or an overestimate of contaminated areas/unnecessary remedial action, respectively. Laboratory analytical errors will be controlled by establishing appropriate controls for data quality (*e.g.*, initial and continuing calibration

verification standards, internal standard and surrogate recoveries, laboratory control samples, as appropriate for each analysis) and validating the resultant data to evaluate potential bias. The project team will consider the validation results during remedial decision making.

2. Laboratory Analytical Sensitivity. Improper specification of RLs could reduce the usability of the collected data for RI/FS decision making. Required RLs were carefully selected for the dual objectives of human health/ecological risk assessment sampling and examination of the spatial distribution of sediment contamination. Consideration of risk assessment "effects levels" and likely remediation goals, respectively, were the basis of RL requirements.

3. Field Screening Errors. A number of screening analyses are under consideration to locate source areas/"hot spots". Due to uncertainty and potential bias in the field analytical techniques and based on the selected spatial scale of the survey techniques, some contaminant source areas may go undetected. Potential bias in immunoassay screening will be controlled by confirmatory primary analytical methods. In addition, survey efforts will be implemented in an iterative manner (*e.g.*, subsequent surveys will adjust sampling locations and frequency based on the review of the results of the initial survey).

4. Sediment Core Sampling Density. The proposed size of the sediment core sample population must be adequate to characterize the Study Area. During design of the 2006 Low Resolution Sediment Coring Program, USEPA Decision Error Feasibility Trials (DEFT) software will be used to evaluate the necessary sample population to provide acceptable percentages of Type I (false positive) and Type II (false negative) errors, considering the statistical distribution and variance of the historic data set. This evaluation will be updated after implementation of the low resolution sediment coring program to establish a basis for potentially required data gap coring efforts.

5. Modeling Errors. Potential errors in the Passaic River/Newark Bay numerical modeling will impact remedial decision making. For example, errors in the rates selected for sediment deposition and/or scour could lead to inappropriate conclusions regarding the potential burial of contaminated sediments, possibly causing inadequate remediation. Modeling errors will be controlled by evaluating direct measurements of parameters whenever possible (such as evaluation of depositional chronology from high resolution sediment cores and SedFlume testing) and by testing the model's skill at prediction of known parameters. The nature of future development in the Study Area may also impact the effectiveness of the model's predictions (70-year prediction to be examined). To control this source of error, data gathered via WRDA real estate and socioeconomic investigations will be assessed to characterize likely future development in and around the Study Area.

6. Geophysical Survey Error. The geophysical data from the side scan sonar (SSS) and sub-bottom prove-out will be evaluated by an experienced marine geophysicist to assess the utility of the obtained data. If the geophysical methods are not found to be applicable for the Lower Passaic River, alternate methods will be evaluated to address the associated study questions (*e.g.*, magnetometer and/or underwater camera surveys may be implemented to identify debris targets that could impact dredging feasibility)

and/or the study questions will be fulfilled to the greatest extent possible by other programmed investigations (*i.e.*, if sub-bottom surveys are not found to be useful, the physical description of sediment stratigraphy will be assessed primarily through examination of sediment cores).

7. Errors in Mass Balance/Evaluation of External Loads. Potential errors in estimates of external contaminant loads to the system will result in errors/uncertainty in the contaminant mass balance and remedial decision making for the Study Area. For example, combined sewer overflow (CSO) sampling during storm events may not adequately represent unknown and intermittent industrial discharges. The sampling design will be optimized, where possible, to obtain the most representative samples, and in this example, it may be possible to sample sludge within the combined sewer system to attempt to further characterize the spectrum of contaminants/discharges present in the system. Errors will also be controlled by iterative sampling events and by considering each line of evidence (results of CSO, water column, and sediment sampling events) that address the potential impacts of point source discharges within the Study Area.

8. Errors/Uncertainty in Risk Assessment. If risks associated with site-related exposures are overestimated (*i.e.*, false positive), a potential consequence is unnecessary remedial work that could itself be biologically detrimental. If risks are underestimated (*i.e.*, false negative), a possible consequence is to fail to conclude that remedial action is required, resulting in continuing potential for adverse effects to human and ecological health. To control for these possible errors, exposure parameters will be carefully selected to represent Reasonably Maximally Exposed individuals. The Trustees' natural resource damage assessment will include site specific studies of injury and exposure, where possible. That information may also be useful in the RI/FS risk assessment to control for errors.

9. Errors/Uncertainty in Remedial Alternative Performance Data. The comparison of remedial alternatives for the FS effort requires the assessment and weighting of remedial alternative performance data (*e.g.*, ex-situ treatment cost per ton, percent reduction in contaminated volume). This data is primarily obtained from literature, seminar presentations, and interviews with USEPA and other agency project management staff. Errors in reported performance data will skew the comparative evaluation of alternatives and could lead to a less than optimal recommended alternative. Decision errors will be controlled by conducting a literature survey to identify and compare multiple sources of performance data, where possible, and by considering the findings of Passaic River pilot study efforts conducted by NJDOT-OMR and the USEPA and the In-situ Stabilization Pilot conducted by NJDOT-OMR.

7.0 Optimize the Design for Obtaining Data

The field investigation design, developed to serve RI/FS, WRDA, and NRDA processes, was optimized by developing broad investigation topics, associated subtasks/decision rules, and required tasks/inputs for each of the proposed field investigation and data gathering efforts, are presented in Attachment 1.1 as Tables 1 through 6. The topics and associated tasks were developed to guide the design of the field investigations and ensure

that the effort meets the needs of Steps 2 and 3 of the DQO process, as described above. The information within Tables 1 through 6 is grouped by general categories of data needs, as listed below:

- Table 1 – Site Physical Characteristics.
- Table 2 – Nature and Extent of Contamination.
- Table 3 – Human Health Risk Assessment.
- Table 4 – Ecological Risk Assessment.
- Table 5 – Evaluation of Remedial Alternatives.
- Table 6 – WRDA Restoration Efforts.

TABLE 1. SITE PHYSICAL CHARACTERISTICS

	BROAD TOPICS	SUB-TOPICS and DECISION RULES	TASKS and INPUTS
	What are the hydrologic, hydrodynamic, and sediment transport characteristics of the Study Area? How can these characteristics support the development of a hydrodynamic, sediment transport, and contaminant fate and transport model?	1. What are the major hydrodynamic and hydrological factors that affect the distribution of the COPCs and COPECs? Decision Rule: Sufficient data is to be collected such that the hydrodynamic model can be calibrated and validated.	1A. Baseline, fixed-point, time series water column data (*e.g.*, water levels, temperature, and salinity) for calibration of the hydrodynamic components of the model. TSS, POC, DOC, and grain size measurements under varying tidal conditions, upstream river discharge, and stratification.
			1B. Water quality data collected from instruments installed on permanent moorings, including current velocity data from Acoustic Doppler Current Profilers (ADCP), conductivity and temperature data from probes, and turbidity data from Optical Backscatter Sensors (OBS).
			1C. Results of conductivity, temperature and depth (CTD) surveys (salinity, temperature, and pressure data) supplemented by sampling for suspended sediment concentration, total dissolved salts, conductivity, POC, grain size, TSS, and VSS. Vertical profile data collected at NJDOT-OMR mooring sites including TSS, total dissolved salt, conductivity, and water density. Vertical profile data collected at Superfund mooring sites for TSS, VSS, and conductivity.
			1D. Results of detailed tidal cycle surveys (including dye studies) conducted by NJDOT-OMR in the Harrison Reach to characterize the spatial structure of currents, stratification, and bottom shear stress in the vicinity of the pilot dredging study area, supplemented by water sampling for TSS, dissolved salt, conductivity, and grain size. Results of Superfund cross-sectional surveys at neap and spring tides supplemented by water sampling for TSS, VSS, conductivity, and grain size.
			1E. USGS characterization of surface water above the Dundee Dam for TSS, VSS, grain size of suspended solids in water samples, POC and Be-7. Data from flow gauges at Dundee Dam. Information on loads from the Contaminant Assessment and Reduction Program (CARP) database. Refer also to Contaminant Mass Balance in Table 3.
		2. What control structures (*e.g.*, dams, locks, tide gates) are present in the Passaic River and adjacent waterways and how do they need to be considered in hydrodynamic evaluations/ modeling efforts? Decision Rule: The function/effects of control structures identified in the Study Area must be appropriately accounted for in the hydrodynamic and sediment transport models.	2A. Identify control structures, if present.
			2B. Evaluate effects of control structures on study area, if applicable.
		3. How will sediment erosion and depositional mechanisms (including storm events and tidal influences) in the Passaic River affect the fate and transport of contaminated sediment, COPCs, and COPECs (*e.g.*, will burial of contaminated sediment by new sediment impact recovery/natural attenuation)? What are the geotechnical properties of sediments in the Lower Passaic River and its tributaries, adjacent waterways (*e.g.*, Hackensack River) and their tributaries, Newark Bay, and flood plain areas? Decision Rule: Sufficient data is to be collected such that the sediment transport model can be calibrated and validated	3A. Grain size distribution (sieve and hydrometer analyses; Laser In-Situ Scattering and Transmissometry (LISST); Malverne Mastersizer), bulk density, dry density, porosity, organic carbon content from sediments of the Passaic River and its tributaries, adjacent waterways and their tributaries, Newark Bay, and the floodplain. Sediment samples are to be collected during geophysical surveying and/or low resolution
			3B. Bed properties of Passaic River and its tributaries, adjacent waterways and their tributaries, Newark Bay, and floodplain areas from historic data and RI/FS sampling programs, including sediment sample analyses and geophysical surveys.
			3C. Soil geotechnical properties in riverbank areas.
			3D. Sediment and erosion depositional mechanisms from dredging pilot study results.
			3E. Location and depth to sediment from bathymetric survey, results of radiological analysis of surface sediment samples for Be-7, characterization of recent sedimentation rates and patterns using Cs-137 and Pb-210 profiles, sediment properties (organic carbon, bulk density, moisture content); evaluation of sediment erosion rates using SedFlume and Gust Microcosm erosion testing devices, evaluate in-situ settling/flocculation of sediment using a Modified Valeport Settling Tube, LISST/OBS and a video settling tube.
	What are physical features of the Study Area, including upland topography, river bathymetry, stratigraphy, and habitat?	4. What is the bathymetry of the Lower Passaic River and its tributaries, adjacent waterways and their tributaries, and Newark Bay? What is the utility of geophysical investigations (SSS and sub-bottom profiling) in the Lower Passaic River for identification of sediment type, stratigraphy, and debris targets? Decision Rules: • If comparison of historic bathymetric data to 2004 data indicates significant changes in river bed elevation (≥2 feet), the usability of historic sediment data will be qualified appropriately and the design of the Low Resolution Coring Program adjusted accordingly. • If review of geophysical data from the SSS and/or sub-bottom prove-out is deemed usable by a marine geophysicist, appropriate geophysical surveys will be extended over the full Study Area, to the extent practical. • If surface sediment type mapping obtained from the SSS survey correlates with chemical data on the extent of COPCs and COPECs, the mapping will be used as an additional line of evidence for the determination of the horizontal extent of contaminated sediment. • If subsurface sediment stratigraphic mapping obtained from the sub-bottom survey correlates with chemical data on the extent of COPCs and COPECs, the mapping will be used as an additional line of evidence for the determination of the vertical extent of contaminated sediment.	4A. Bathymetric survey data and mapping in hardcopy and electronic formats, including USACE and TAMS 2004 data and digitized (not scanned) versions of USACE 1989, Tierra Solutions, Inc. (TSI) 1999, and TSI 2000 bathymetric surveys.
			4B. Identification of potential deposition and scour areas.
			4C. Identification of potential bathymetric changes associated with historic storms (*e.g.*, Hurricane Floyd), based on comparison of TSI 1999, TSI 2000, and USACE 2004 bathymetric survey data.
			4D. SSS and sub-bottom survey data from a limited number of "prove-out" locations.
			4E. "Ground truth" sediment near-surface cores and deep cores for calibration of the SSS and sub-bottom data, respectively and collection of sediment geotechnical data.
			4F. If SSS is implemented, the texture of surficial sediments (*e.g.*, ripple patterns, debris patterns).
			4G. If SSS is implemented, the amount/extent of debris and other targets (*e.g.*, utilities, wrecks) in the Passaic River for evaluation of the feasibility of remedial dredging and the feasibility of achieving restoration objectives at a particular site.
			4H. If sub-bottom surveying is implemented, the sediment stratigraphy below the Study Area riverbed.
		5. What are the physical features and topography of upland project areas adjacent to the Lower Passaic River, including the [10, 20, 100] -year flood plains? What is the wetland boundary in the Meadowlands? Decision Rule: Obtain survey data and mapping to adequately characterize the Study Area for RI/FS preparation.	5A. Land surveying and aerial photography field data.
			5B. Topographic maps at 1 inch = 30 ft scale that meet ASPRS Class 3 Map Accuracy for investigation planning and subsequent visual presentation of RI/FS data.
			5C. Shoreline and planimetric electronic data in AutoCAD and ArcGIS electronic formats.
			5D. Land use, vegetation types, urban characteristics, etc. of floodplain area adjacent to the Passaic River and its tributaries, adjacent waterways and their tributaries, and Newark Bay.
		6. What cultural resources, or significant or unique habitats and communities might be disturbed by remedial action (*e.g.*, submerged aquatic vegetation, wetlands, threatened or endangered species)? Decision Rule: Adequate data will be obtained on the presence/absence of cultural resources and significant or unique habitats and communities to assess their impact on remedial implementation and feasibility.	6A. Identification of significant cultural resources in the Study Area.
			6B. Delineation and assessment of submerged aquatic vegetation (SAV), wetland, and shoreline habitats.
			6C. Identification of threatened or endangered species or unique communities/populations.

TABLE 2. NATURE AND EXTENT OF CONTAMINATION

	BROAD TOPICS	SUB-TOPICS and DECISION RULES	TASKS and INPUTS
	What are the COPCs and COPECs in the Study Area environmental media? What is the current spatial distribution of COPCs and COPECs concentrations in the river sediments, both horizontally and vertically?	7. What is the current inventory of COPCs and COPECs in the river? What fraction of this inventory is or will become available over time? What is the most upstream point potentially impacted by contaminants released in the saline (brackish) portion of the estuary? What is the potential contribution of this inventory to the harbor and Newark Bay? Decision Rules: • Contaminants will be identified as COPCs if they meet the criteria in Section 5.1 of the PAR. • Contaminants will be identified as COPECs if they meet the criteria in Section 6.1 of the PAR. • Estimated availability of inventory and upstream transport to be evaluated via Passaic River/Newark Bay model output.	7A. VOC, SVOC/PAH, pesticide, inorganic, AVS/SEM, dioxin/furan, PCB congener, and PCB Aroclor concentrations in surface and subsurface sediments, as determined via RI/FS low resolution and high resolution sediment coring programs. Some sampling locations to be co-located with geotechnical samples collected to characterize sediment bed properties (refer to Task 3A). Frequency of detection of each parameter.
			7B. VOC, SVOC/PAH, pesticide, inorganic, dioxins/furans, and PCB congener surface water concentrations from RI/FS water column sampling (*e.g.*, data from moorings, small volume composite grab samples, large volume samples, and SPMD). The collected samples should be coordinated with other surface water quality measurements such as TSS analyses (refer to Tasks 1A through 1E). Frequency of detection of each parameter.
			7C. Historical sediment and water quality data.
			7D. Passaic River/Newark Bay model runs to evaluate availability and transport of contaminant inventory over time.
			7E. Risk-based criteria and/or preliminary remediation goals (PRGs), lists of Class A carcinogens, etc.
		8. What is the horizontal and vertical extent of the contaminated sediments (unacceptable COPC and COPEC concentrations) in the Study Area? Decision Rule: Contaminant concentrations exceeding project-specific action levels (to be determined) will be geostatistically analyzed along with sediment type data from geotechnical and geophysical surveys to establish the extent of contaminated sediments requiring remediation.	8A. Data from "Identify COPCs/COPECs" (Tasks 7A through 7E above).
			8B. Results of screening investigations (*e.g.* immunoassay sediment analyses) that employ rapid field surveys of water and sediment quality to identify the locations of potential contaminated sediment deposits and target these areas for subsequent low resolution sediment coring.
			8C. "Data gap" low resolution sediment coring results based on geostatistical and judgmental sampling based on data from Task 8B.
			8D. Comparison of historic and current bathymetric mapping to identify whether storm events or other mechanisms (*e.g.*, Hurricane Floyd of 1999) redistributed contaminated sediments, necessitating recharacterization of previously sampled areas.
			8E. Historical sediment characterization data that meet project quality standards and are deemed to be representative of current conditions (evaluation criteria to include review of co-located low resolution sediment core sample data).
			8F. A description of contaminated sediment depositional chronology from the high resolution sediment coring program. Radionuclide dating results from finely segmented cores. Chemical concentration data from selected high resolution sediment core segments based on radionuclide dating.
			8G. Low resolution sediment core and mudflat sediment core results for geostatistical and/or other spatial analyses.
			8H. Maps of sediment physical properties (*e.g.*, grain size, geologic description, stratigraphy from core descriptions and sub-bottom profiling, if applicable) where field data indicate a correlation between contamination and specific physical properties (such as fine-grained sediments) based on Tasks 4A through 4H.
	What are the major sources and processes controlling COPC and COPEC distribution in the Lower Passaic? What is the COPC and COPEC mass balance?	9. What are the major external sources of the COPCs and COPECs to the Lower Passaic? • What are the loads at the Dundee Dam? • What are the loads contributed by the tributaries? • What are the loads contributed by CSOs and sewer discharges? • What are the loads contributed by direct industrial discharges? • What are the magnitude and the direction of the net tidal transport in the river? • What is the magnitude of gas exchange and dry and wet atmospheric deposition? • What are the magnitude and the direction of the net ground water transport in the river? • What is the distribution of the particulate and dissolved phases in the water column? Decision Rules: • Sufficient data will be collected to characterize contaminant loads at Study Area boundaries. • Sufficient data will be collected to characterize discharges (*e.g.*, • Sufficient data will be collected to characterize other sources of c	9A. Results from water column monitoring (e.g., small volume composite grab samples, large volume samples, and SPMD).in the Lower Passaic River; at boundaries with tributaries, Newark Bay, and the Hackensack River. (refer also to Task 1A). CSO and wastewater treatment plant (WWTP) sampling efforts (to be conducted by others).
			9B. Results of hydrogeological investigations and modeling.
			9C. Results of atmospheric deposition investigations including wet and dry deposition, emission records, and air-water interface concentrations for estimating deposition/volatilization.
			9D. Completion of the preliminary mass balance calculations and sensitivity analyses.
		10. What are the major internal processes affecting COPCs and COPECs? • What are the contributions of sediment resuspension and deposition (from storms, bioturbation, tidal action, etc.), adsorption and desorption, porewater diffusion and porewater displacement (groundwater movement)? • What other in-river processes may be important (photolysis, hydrolysis, precipitation, biodegradation, weathering)? Decision Rules: • Sufficient data will be collected to characterize contributions to water column contamination due to bioturbation and porewater releases. • Calibrated and validated model output will be used to forecast the impacts of other in-river processes on COPCs and COPECs.	10A. Results of bioturbation sampling, porewater sampling (*e.g.*, "peepers"), and hydrogeological investigations (see Task 9B).
			10B. Results of model output regarding sediment transport associated with storm events, tidal action, etc. and the impacts of other in-river processes on the fate and transport of COPCs and COPECs.
		11. How have the external and internal sources varied over time and how are they likely to vary in the future? How will external loads be expected to vary? What factors govern the internal loads and how will these vary? Decision Rule: Sufficient data will be collected to characterize internal and external sources and loads to calibrate the hydrodynamic and sediment transport models and prepare a cohesive geochemical evaluation of the Study Area.	11A. Depositional chronology data from high resolution sediment coring program.
			11B. Low resolution sediment coring analytical data, water column sampling analytical data, and Passaic River/Newark Bay model output.
			11C. Historic data from literature regarding sources and characterization of contaminant loads. Evaluation of historic data via calculation of ratios between various contaminants, PCB congeners, and dioxins; reconciliation of unique contaminant signatures, water column concentrations, and solids transport data for various sources (*e.g.*, tributaries, discharges).
		12. What is the rate at which each COPC/COPEC attenuates (including biodegradation and weathering mechanisms), is exported, or becomes unavailable from locations along the river? Decision Rule: Geochemical evaluation of RI/FS and historic data, information from the literature, and calibrated model output will be used to evaluate the potential for natural attenuation of COPCs and COPECs.	12A. Evaluation of sediment and water column analytical data for evidence of biodegradation and natural attenuation mechanisms and contaminant breakdown products.
			12B. Literature information on COPC and COPEC natural attenuation and biodegradation.

	BROAD TOPICS	SUB-TOPICS and DECISION RULES	TASKS and INPUTS
TABLE 3. HUMAN HEALTH RISK ASSESSMENT	What is the current and future human health risk associated with exposure to sediment, surface water, and/or consumption of edible portions of fish or shellfish? (Potential risks for consumption of other species (*e.g.*, waterfowl) will be evaluated qualitatively.	13. Are the environmental data for sediment, surface water, and biological tissue of acceptable quality for use in estimating human health risks? Decision Rule: Based on the outcome of the data usability evaluation, retain those data determined to be of acceptable quality for use in risk assessments, otherwise eliminate. For retained analytical results, if data sets are comparable (based on criteria specified in the data usability evaluation), then combine for use in risk assessment; otherwise, select the subset(s) that best meet DQOs.	13A. Evaluate data usability of relevant environmental media including quality of data with respect to: sample quantitation limits, qualifiers and codes, blanks, and tentatively identified compounds (TICs). Evaluate data comparability by examining analytical methods, QA/QC procedures, and similarity of results.
		14. Is the spatial coverage of COPCs adequate to quantify human health exposures with a specified level of confidence? Decision Rule: If the spatial coverage of risk assessment data within each defined area/habitat is adequate to meet the objectives (with respect to spatial and statistical requirements) developed during the sample design phase, then calculate exposure point concentrations (EPCs); otherwise, collect additional analytical data to address data gaps.	14A. Evaluate adequacy of spatial coverage within each exposure area/unique habitat with respect to sampling needs identified in the sample design phase.
		15. Do current or projected future COPC concentrations in sediments from the Passaic River pose an unacceptable health risk [exceeding the NCP risk range defined as a cancer risk >1E-04 to 1E-06 and/or a non-cancer hazard index (HI) >1] to human receptors? Decision Rule: If estimated cumulative human exposure results in an unacceptable health risk (*i.e.*, a cancer risk >1E-06 and/or a non-cancer HI>1), then further evaluation of remedial options or restoration will be considered as part of the FS process.	15A. Identify appropriate exposure scenarios and population groups based on the human health conceptual site model. 15B. Identify COPCs in sediment and water based on a risk-based contaminant screening process. 15C. Calculated potential carcinogenic risks and noncarcinogenic hazard indices for direct exposures to sediment. Cancer risks and hazard indices will be calculated for both current and predicted future conditions. Calculations will be based on concentrations of COPCs in surface sediments and water from the Passaic River. Concentrations may be based on: (a) current analytical measurements for surface sediments; (b) current analytical measurements for sediment at depth that may be exposed in the future; (c) results of sediment modeling exercises. 15D. Emerging chemicals of potential concern as identified by USEPA will be considered as COPCs in the Study Area.
		16. Do current or projected future COPC concentrations in tissues of fish and shellfish from the Study Area pose an unacceptable health risk (defined as a cancer risk >1E-06 and/or a non-cancer HI>1) from consumption by human receptors? Decision Rule: If estimated cumulative human exposure results in an unacceptable health risk (*i.e.*, a cancer risk >1E-06 and/or a non-cancer HI>1), then further evaluation of remedial options or restoration will be considered as part of the FS process.	16A. Identify appropriate exposure scenarios and population groups. 16B. Identify COPCs in edible portions of fish and shellfish based on a risk-based contaminant screening process. 16C. Determine appropriate site-specific exposure factors. 16D. Calculate potential carcinogenic risks and noncarcinogenic hazard indices for direct exposures to consumption of fish and shellfish. Risk and hazard indices will be calculated for both current and predicted future conditions. Calculations will be based on concentrations of COPCs in edible fish and shellfish tissue. Concentrations may be based on: a) current analytical measurements for fish and shellfish species collected from the Study Area; b) estimated tissue concentrations based on food web modeling using current or predicted sediment concentrations.
		17. Do current or projected future COPC concentrations in tissues of potential edible species (*e.g.*, waterfowl) from the Study Area pose an unacceptable health risk from consumption by human receptors? Decision Rule: If qualitative evaluation of exposure and risk indicate a potential for unacceptable health risk, then further evaluation of remedial options or restoration will be considered as part of the FS process.	17A. Identify appropriate exposure scenarios and population groups. 17B. Identify possible COPCs in edible portions of other species (*e.g.*, waterfowl) based on potential for bioaccumulation. 17C. Evaluate potential for exposure and risk qualitatively.

TABLE 4. ECOLOGICAL RISK ASSESSMENT

	BROAD TOPICS	SUB-TOPICS and DECISION RULES	TASKS and INPUTS
	What is the current ecological risk associated with exposure to sediment and porewater and/or consumption of edible portions of fish, shellfish, or other edible species (e.g. waterfowl)?	18. Are the environmental data for sediment, surface water, and biological tissue of acceptable quality for use in estimating ecological risks? Decision Rule: Based on the outcome of the data usability evaluation, retain those data determined to be of acceptable quality for use in risk assessments; otherwise eliminate. For retained analytical results, if data sets are comparable (based on criteria specified in the data usability evaluation), then combine for use in risk assessment; otherwise select the subset(s) that best meet DQOs.	18A. Evaluate data usability of relevant environmental media including quality of data with respect to: sample quantitation limits, qualifiers and codes, blanks, and TICs. Evaluate data comparability by examining analytical methods, QA/QC procedures, and similarity of results.
		19. Is the spatial coverage of COPECs adequate to quantify ecological exposures with a specified level of confidence? What is the biologically active zone? Decision Rule: If the spatial coverage of risk assessment data within each defined exposure area/habitat is adequate to meet the objectives (with respect to spatial and statistical requirements) developed during the sample design phase, then calculate EPCs; otherwise, collect additional analytical data to address data gaps. Evaluate weight of evidence to determine depth of the biologically active zone.	19A. Evaluate adequacy of spatial coverage within each exposure area/unique habitat with respect to sampling needs identified in the sample design phase. 19B. Obtain sediment profile imagery (SPI), conduct preliminary grab sampling for benthic organisms, obtain vertical profile of oxidation-reduction potential in near-surface sediments.
		20. Do current or projected future COPEC concentrations in sediments from the Study Area pose an unacceptable risk to ecological receptors of concern either (a) directly exposed to contaminants in sediment, porewater, and/or surface water or (b) exposed to contaminants through the food web? Decision Rule: For each assessment endpoint, determinations of risk and magnitude of risk (*i.e.*, high or low magnitude) will be provided in the Field Sampling Plan Volume 2. This will also include the process for integrating each line of evidence into the weight-of-evidence process to interpret the risk findings.	20A. Develop Ecological Conceptual Site Model that depicts contaminant sources, potential migration pathways, exposure pathways, and receptors of concern (ROCs). Select ROCs based on degree of contact with sediment/mudflats, dietary preferences, and habitat suitability. Inputs include data from historical and planned habitat population surveys (under WRDA); in addition, consideration to possible restoration objectives that could results in the re-establishment of extirpated populations within the Study Area.
			20B. Identify COPECs by a screening process identified in the PAR. Comparisons of historical, current, and any future contaminant concentrations will be made to COPEC screening benchmarks for both bioaccumulative and non-bioaccumulative contaminants.
			20C. Estimate concentrations of COPECs in surface sediments and porewater from the Study Area. Concentrations may be based on: (a) current/future analytical measurements for surface sediments and porewater; (b) current/future analytical measurements for sediments at depth that may be exposed in the future; and (c) modeling output.
			20D. Estimate concentrations of COPECs in surface water in the Study Area. Concentrations may be based on current/future analytical measurements for water and the results of hydrodynamic modeling.
			20E. Estimate concentrations of COPECs in prey items consumed by upper trophic level ROCs. Concentrations may be based on: (a) current/future analytical measurements of fish and prey species collected from the Study Area; (b) estimated tissue concentrations based on food web modeling using current or predicted sediment concentrations.
			20F. Ecological effects data may be obtained using a variety of methods including, but not limited to, dose-response studies reported in the literature, site-specific laboratory bioassays, and population- and community-level bioassessment studies conducted in the Study Area.
			20G. Quantify risk estimates using hazard ratio methods (*e.g.*, comparison of NOAELs/LOAELs to exposure concentrations).

TABLE 5. EVALUATION OF REMEDIAL ALTERNATIVES

BROAD TOPICS	SUB-TOPICS and DECISION RULES	TASKS and INPUTS
What is the optimal remedial alternative to address unacceptable human health and/or ecological risks at the Study Area?	21. Has sufficient data been collected to comparatively evalute remedial alternatives, including no action, monitored natural recovery, removal, in-situ treatment, or capping? What interim remedies are desirable and feasible (if any)? Sufficient data collection is initially defined as fulfilling the tasks/inputs identified in Tables 1-6 inclusively, which are intended to lead the project to the FS stage. Additionally, it is envisioned that the stakeholders will identify and rank the decision criteria for FS alternative evaluation through meetings of the Remediation Options Workgroup. The identified decision criteria may prompt amendment of the DQOs and additional data collection necessary to complete the FS evaluation. Decision Rule: Applicable remedial options (including no action) will be comparatively evaluated according to the CERCLA evaluation criteria and assigned weightings. The remedial alternative with the most favorable combined weighting will be recommended for implementation.	21A. Contaminant concentrations from historic data and RI/FS field investigations, horizontal and vertical extent of contamination, and extent of contaminant migration (including an evaluation of sediment stability). 21B. Dredge performance and monitoring data from the Environmental Dredging Pilot Study and data obtained from literature searches. 21C. Treatability data from the Passaic Sediment Decontamination Technology Pilot, NY/NJ Harbor Sediment Decontamination Program, OMR In-situ Stabilization/Deep Soil Mixing Pilot Studies and data obtained from literature searches. 21D. Performance criteria for other in-situ/ex-situ treatment alternatives proposed to reduce the toxicity, volume, or mobility of sediment contaminants. 21E. Material handling and physical properties of contaminated sediments from the Passaic River in regard to sediment dewatering and treatment issues, from geotechnical and geophysical programs.
	22. How will the presence of debris, cultural resources, recreational resources, sensitive habitats, the volume and extent of contaminated sediment, and the physical/geotechnical and chemical properties of the contaminated sediment impact the feasibility of dredging and other remedial alternatives? Decision Rule: The amount and nature of debris and sediment geotechnical properties will be considered to evaluate the implementability of a dredging alternative.	22A. Debris assessment from SSS and potentially a magnetometer survey. 22B. Location and type of cultural resources and sensitive habitats from Task 6. 22C. Volume and extent of contaminated sediment and sediment properties from Task 8 and Task 21 above. 22D. Assessment of recreational resources that could be disturbed by remedial action.
	23. What is the forecasted reduction in human and ecological risk for various remedial alternatives (e.g., minimization of contaminant export from a particular location), including interim remedies, and over what future duration? Decision Rule: The estimated reduction in risk for each remedial alternative evaluated will be considered as part of the assessment of short-term and long-term effectiveness of the alternative.	23A. Human and ecological risk assessments for various remedial scenarios.
	24. Will contaminant loading to and from sources outside the Lower Passaic River recontaminate the Passaic River to an unacceptable level following a potential sediment remediation action in the Passaic River? Decision Rule: Model output will be used to estimate the potential for recontamination of remediated portions of the Study Area due to external loads. Projections of potential recontamination will be weighed in the evaluation of remedial alternatives.	24A. Mass balance data and characterization of external contaminant loads to the Study Area from Task 9.
	25. How will the availability of disposal sites/placement sites (*e.g.*, upland sites, CDFs) and their acceptance criteria impact the feasibility of remedial dredging? Is decontamination and production of beneficial use products an option? Decision Rule: The availability of dredged sediment disposal sites and availability of decontamination/reuse facilities will be considered during assessment of the implementability of a dredging/sediment removal alternative.	25A. Telephone and literature survey of CDF status, permit acceptance criteria, treatment types available and performance data. Telephone and literature survey of facilities that can produce beneficial use products.
	26. What are the Resource Conservation and Recovery Act (RCRA) disposal characteristics of contaminated sediments from the Passaic River? Decision Rule: Sediment analytical results will be compared to RCRA action levels for characteristics of toxicity, reactivity, corrosivity, ignitability and other disposal criteria. Assessment of disposal characteristics will be used to evaluate implementability and estimated cost of remedial alternatives.	26A. Toxicity characteristic leaching procedure (TCLP) extract concentrations from sediment samples. 26B. Geotechnical and wet chemical analyses including moisture content, TOC, and paint filter test analyses. 26C. Survey of currently available and potential future dredged sediment disposal sites from Task 25.

TABLE 6. WRDA RESTORATION EFFORTS

	BROAD TOPICS	SUB-TOPICS and DECISION RULES	TASKS and INPUTS
	What is the suitability of candidate sites for WRDA restoration efforts? Collect data needed to support development of a restoration project concept design and analysis via environmental investigations, habitat evaluation procedures, hydrogeomorphic approach, and rapid bioassessment protocols.	27. Do the detected concentrations of chemical contaminants in the candidate restoration site environmental media exceed NJDEP Technical Site Remediation Standards, reference values, and/or other Applicable or Relevant and Appropriate Requirements (ARARs)? Are the detected concentrations of contaminants likely to have an adverse impact on site restoration (*e.g.*, plantings, biota)? Decision Rule: The detected concentrations of environmental contaminants at candidate sites will be considered in the prioritization of sites for WRDA restoration efforts. The following categories of restoration opportunities are envisioned: • Clean sites removed from future influence of river contamination (*e.g.*, upland or upstream site) that can be "fast-tracked" for restoration. • Isolated contaminated sites that have a remediation phase, but which is independent of remedial action for Study Area (*e.g.*, contaminated upland site). • Contaminated sites dependent on the Study Area remedy (restoration to be implemented post Study Area remediation).	27A. TCL/TAL (PAHs only for semivolatile fraction), cyanide, PCB congener, and dioxin/furan concentrations in surface and subsurface soils and sediments. 27B. TCL/TAL (PAHs only for semivolatile fraction), cyanide, PCB congener, dioxin/furan, and TOC concentrations in groundwater and surface water. 27C. NJDEP Site Remediation Criteria, ecotoxicological benchmarks, reference values, and ARARs for evaluation of environmental media analytical results 27D. Candidate Site Restoration chemical screening criteria, consisting of ecological risk-based action levels for adverse impacts on biota and plantings associated with proposed restoration plan
		28. What is the appropriate restoration design for suitable candidate sites (*e.g.*, horticultural design and planting, aesthetics, channel layout) based on site-specific findings? Decision Rule: Sufficient data on site physical features will be collected to support the development of an appropriate restoration design.	28A. Elevations and topographic features of the candidate restoration sites from land surveying and aerial photography field activities. 28B. Geotechnical properties of candidate site soils/sediments to support restoration feasibility analyses. 28C. Grades of the side slopes of the Passaic River and/or its tributaries at candidate restoration sites (for possible design of bank stabilization/regrading measures associated with restoration). 28D. Site access characteristics and the locations of utilities and other features. 28E. Topographic maps at 1 inch = 30 ft scale that meet ASPRS Class 3 Map Accuracy. 28F. Shoreline and planimetric electronic data in AUTOCAD and ARCGIS electronic formats. 28G. Characterization of groundwater and surface water elevations, fluctuations, and flow directions/regimes to understand the hydrologic factors that may affect restoration feasibility analyses. 28H. Assessment of cultural resources present at candidate restoration sites that could be disturbed by rehabilitation efforts. 28I. Characterize the socioeconomic characteristics of the Passaic River watershed area to support WRDA candidate restoration site decision making. 28J. Evaluate the real estate characteristics of the Passaic River watershed area to support WRDA candidate restoration site decision making. 28K. Determine consistency with NRDA requirements. 28L. Other National Environmental Policy Act (NEPA) Environmental Impact Statement (EIS) data needs.
	Identify and evaluate the feasibility of other WRDA projects in the Study Area.	29. Is there a quantifiable/defensible benefit to conducting additional sediment remediation (beyond what is required under CERCLA) through a WRDA contribution to the remedial effort?	29A. The results of the comparative evaluation of remedial alternatives from Tasks 21-26. 29B. Ecological risk assessments for potential WRDA expanded remediation scenarios. 29C. Economic analysis of the proposed project.
		30. To what extent are Passaic River remedial actions warranted/feasible to reduce the export of contamination to other areas in the Hudson Raritan Estuary, even if recontamination of the Passaic River sediments may be experienced due to uncontrolled upstream sources? Decision Rule: The implementation cost for a remedial alternative to improve dredged material management for the navigational dredging program will be evaluated via an economic analysis.	30A. Results of evaluation in Task 24. 30B. Model output to predict fate and transport of contaminants from external loads following Study Area sediment remediation, transport to Newark Bay, and durations associated with recontamination of the Study Area. 30C. Economic analysis of avoided navigational dredging and disposal costs in Newark Bay maintenance and deepening projects.

Attachment 1.2

Data Needs/Data Use Table

Data Needs/Data Uses Table for Fall 2005 Program

Data Need	Data User	Program	Medium	Parameter	Methodology/ Protocols	RLs	Rationale for Analysis	Notes (e.g., sample quantity, distribution)	Data Use
Radionuclide activities and contaminant concentrations in finely segmented sediment cores	Geochemist, Modeler	High Resolution Coring Program	Sediment	a) Be-7, Cs-137, Pb-210	QAPP Table 4-4	QAPP Table 2	• Determine age of deposition (different species account for different time frames) • Determine type of depositional environment (marine/terrestrial)	The 2005 High Resolution Coring Program includes 8 high resolution cores (refer to FSP for locations and rationale). The cores will be initially segmented into small slices for initial radionuclide dating. Following review of the radionuclide profiles, selected segments and/or composite of multiple segments will be submitted for chemical analysis.	Depositional chronology for contaminants; update of Conceptual Site Model; investigation of historic sources and loads.
				b) PCB Congeners	Method 1668A		• Identify and characterize the contaminants • Determine extent of contamination • Determine the rate of contaminant declination • Establish diagnostic fingerprint of source(s) over time • Determine extent of diagenesis		
				c) Dioxins/furans	Method 1613B				
				d) Pesticides (including DDT and metabolites)	Modified Method 8081 (GC-MS-SIMs or GC-ECD)				
				e) PAHs	Modified Method 8270 (GC-MS-SIMs)				
				f) TAL Metals	CLP ILM0.5.3 with flex clause				
				g) Total Organic Carbon	Lloyd Kahn		• Determine mobility of certain contaminants • Control sediment erodibility		
				h) Grain Size	ASTM D422 or ASTM D4464		• Establish nature of the sediment material • Evaluate relationship between partilce size and contaminant concentration • Determine type of depositional environment (comparing normal flow events to flood events)		
				i) Bulk Density	Processing Facility Measurement		• Estimate contaminant mass present in sediments • Evaluate remedial option feasibility • Estimate total mass of contaminants present in study area		
				j) X-Radiograph	Field Test		• Characterize sediment transport and erosion • Used to scale the degree of mixing/bioturbation • Examine density variations that may be indicative of major hydrologic/depositional events		

Data Needs/Data Uses Table for Fall 2005 Program

Data Need	Data User	Program	Medium	Parameter	Methodology/ Protocols	RLs	Rationale for Analysis	Notes (e.g., sample quantity, distribution)	Data Use
Sediment contaminant concentrations and geotechnical properties	Remedial Engineer, Modeler, Risk Assessor	Low Resolution Coring Program	Sediment	a) Be-7, Cs-137, Pb-210	QAPP Table 4-4	QAPP Table 2	• Use sediment dating as a screening tool for chemical analysis, based upon High Resolution Program findings	The 2005 Low Resolution Coring Program will consist of 51 cores throughout the Study Area (refer to FSP for locations and rationale). Additional low resolution cores will be added in 2006 based on geostatistical analyses and data gap evaluations.	Contaminant spatial extent (distribution and concentration in sediments); mixing zone depth; sediment transport modeling; sediment material handling properties with respect to remedial alternative evaluation; ecological risk assessment.
				b) PCB Congeners	Method 1668A		• Characterize exposure to co-planar PCBs with "dioxin-like" effects • Provide an accurate method for quantifying total PCB exposures and for source discrimination		
				c) Dioxins/furans	Method 1613B		• Necessary to investigate spatial extent of dioxin/furan contamination		
				d) Dioxin/PCB TEQ Immunoassay	Method 4025		• Plot study of immunoassay test to determine applicability for Passaic River Sediment (Immunoassay data will be correlated with Dioxin/furan (1613B) and PCB Congeners (1668A) data to establish immunoassay as screening tool for Passaic River Sediment)		
				e) PCB Aroclors	GC-MS-SIM (CLP SOM1.0) with flex clause		• Proposed as a lower cost (compared to full congener analysis) alternative for spatial delineation of PCB sediment contamination		
				f) TCL Volatile Organics			• Investigate presence/absence and spatial extent of preliminary COPCs/COPECs in sediment from key human health and ecological exposure areas • Finalize COPC/COPEC selection during baseline risk assessments • Select appropriate bioassessment data sampling locations for FSP • Identify additional sediment sampling needs		
				g) TCL Semivolatile Organics (including PAHs)					
				h) Pesticides (including DDT and metabolites)	Modified Method 8081 (GC-MS-SIMs or GC-ECD)				
				i) TAL Metals	CLP ILM0.5.3 with flex clause				
				j) TPH	NJDEP Method				
				k) Chlorinated Herbicides	Method 8151A				
				l) Methyl-mercury	Method 1630		• Determine the toxic extent of the mercury (methyl-mercury is the most toxic form of mercury)		
				m) Arsenic speciation	Method 1632A		• Determine the mobility of the arsenic • Determine presence/absence and appropriate toxicity as necessary		
				n) Hexavalent Chromium	Method 7199/3060A		• Determine the toxic extent of the chromium (chromium VI is the most toxic form of chromium) • Determine presence/absence and ratio of hexavalent chromium to total chromium for identifying toxicity, as necessary		
				o) Acid Volatile Sulfide/SEM Metals	Method 821-R-91-100/SW-846		• Used to estimate bioavailability of divalent cationic inorganics • Necessary for mercury modeling. Methylation of mercury is related to sulfide production.		
				p) Kjeldahl Nitrogen	Method 351.3		• Determine type and source of nitrogen contamination (Total Kjeldahl Nitrogen = organic nitrogen + ammonia)		
				q) Total Organic Carbon	Lloyd Kahn		• Determine mobility of certain contaminants • Estimate sediment erodibility		
				r) Butyl tins	Lab-prepared SOP		• Identify as preliminary COPC/COPEC in PAR following standard screening criteria (additional characterization necessary to more accurately quantify exposures)		
				s) Cation Exchange Capacity	Method 9081		• Determine the mobility of metals		
				t) Grain Size	ASTM D422		• Sediment physical properties investigated for evaluation of remedial options (e.g., dredging technologies) and sediment transport modeling • Use grain size and pH data to characterize microhabitat conditions during receptor selection and exposure pathway analysis in the Baseline Risk Assessment (BERA)		
				u) Percent Moisture	ASTM D2974				
				v) Atterberg Limits	ASTM D4318				
				w) Specific Gravity	ASTM D854				
				x) pH	Method 9045C				
				y) Bulk Density	Processing Facility Measurement (see FSP for details)		• Estimate contaminant mass present in sediments and evaluation remedial option feasibility • Estimate total mass of contaminants present in study area		
				z) X-Radiograph	Field Test		• Characterize sediment transport and erosion • Used to scale the degree of mixing/bioturbation • Examine density variations that may be indicative of major hydrologic/depositional events		

Data Needs/Data Uses Table for Fall 2005 Program

Data Need	Data User	Program	Medium	Parameter	Methodology/ Protocols	RLs	Rationale for Analysis	Notes (e.g., sample quantity, distribution)	Data Use
				aa) Density Profiler (if equipment becomes available)	Gotthard Density Profiler		• Characterize sediment transport and erosion • Used to scale the degree of mixing/bioturbation • Examine density variations that may be indicative of major hydrologic/depositional events		

Data Needs/Data Uses Table for Fall 2005 Program

Data Need	Data User	Program	Medium	Parameter	Methodology/ Protocols	RLs	Rationale for Analysis	Notes (e.g., sample quantity, distribution)	Data Use
Contaminant concentrations in the water column; evaluate eutrophication component	Geochemist, Modeler, Risk Assessor	Preliminary Water Column Sampling Program	Surface Water (Small Volume Composite Grab Samples)	a) BOD	Method 405.1	QAPP Table 2	• Routine water quality parameter	Small volume water column composite grab samples will be collected.	Assess COPC/COPEC concentrations and transport variation for fate and transport modeling; calibrate eutrophication component of model; human and ecological risk assessment. Since the fate and transport model is carbon-based, the calibration of the eutrophication model affects the fate of the chemicals sorbing into the organic fraction.
				b) COD	Method 410.4		• Routine water quality parameter		
				c) DOC	Method 9060		• Investigate role of organic carbon in water column contaminant fate and transport		
				d) POC	EPA 440.0 or L.Kahn				
				e) TSS	Method 160.2		• Quantify solids present in water column samples to investigate particle-associated transport of water column contaminants		
				f) Trace Mercury & Methy Mercury (whole water & dissolved phase analysis)	Methods 1631 and 1630		• Determine the toxic extent of mercury (methyl-mercury is the most toxic form of mercury)		
				g) TAL Metals (whole water and dissolved phase samples)	CLP ILM0.5.3 with flex clause		• Investigate potential transport of COPCs in the water column • Identify and characterize the contaminants • Determine extent of contamination		
				h) Kjeldahl Nitrogen	Method 351.3		• Determine type and source of nitrogen contamination (Total Kjeldahl Nitrogen = organic nitrogen + ammonia) • Characterize nutrient inputs as stressors for interpreting bioassessment data • Use for calibration and refinement of organic carbon model (primary production model)		
				i) Chlorophyll a	SM 10200-H		• Indicates algal activity in contaminated area (algal blooms)		
				j) Total and ortho-phosphate	Method 365.2		• Use for calibration and refinement of organic carbon model (primary production model)		
				k) Ammonia	Method 350.2		• Use for calibration and refinement of organic carbon model (primary production model) • Routine water quality parameter		
				l) TCL Volatile Organics	GC-MS-SIM (CLP SOM1.0) with flex clause		• Investigate potential transport of COPCs in the water column • Identify and characterize the contaminants • Determine extent of contamination		
				m) TCL Semivolatile Organics (including PAHs)					
				o) Chlorinated Herbicides	Method 8151A				
				p) Dissolved Oxygen	Field Measurement (see FSP for details). pH may be confirmed for some samples in the lab - Method 9045C.		• Routine water quality parameter		
				q) pH			• Routine water quality parameter		
				r)Secchi Depth			• Use for calibration and refinement of organic carbon model (primary production model) • Routine water quality parameters		
				s) Conductivity			• Routine water quality parameter		

Data Needs/Data Uses Table for Fall 2005 Program

Data Need	Data User	Program	Medium	Parameter	Methodology/ Protocols	RLs	Rationale for Analysis	Notes (e.g., sample quantity, distribution)	Data Use
Contaminant concentrations in the water column	Geochemist, Modeler, Risk Assessor	Preliminary Water Column Sampling Program	Surface Water (Large Volume Samples/HOC Sampling Methodology Validation Study)	a) PCB Congeners	Method 1668A	QAPP Table 2	• Investigate transport of PCBs in the water column • PCB congener analyses essential for analytical sensitivity and distinguishing potential multiple sources of contamination	Various methodologies for collecting large volume samples for HOC will be evaluated as described in the FSP. Conventional and hydrodynamic parameters including DO, conductivity, temperature, current, pH will be monitored during the sampling period. A large grab sample will be collected for PAH analysis by modified method 8270 (GC-MS-SIMs). In addition, grab samples will be analyzed for TSS (Method 160.2), POC (L. Kahn), and DOC (Method 9060).	Assess COPC/COPEC concentrations and transport variation for fate and transport modeling; calibrate eutrophication component of model; human and ecological risk assessment. Since the fate and transport model is carbon-based, the calibration of the eutrophication model affects the fate of the chemicals sorbing into the organic fraction.
				b) Dioxins/Furans	Method 1613B		• Investigate transport of dioxins/furans in the water column		
				c) Pesticides (including DDT and metabolites)	Modified Method 8081 (GC-MS-SIMs or GC-ECD)		• Investigate potential transport of COPCs in the water column • Identify and characterize the contaminants • Determine extent of contamination		
			Surface Water (Semi-Permeable Membrane Device Extract)	a) PCB Congeners	Method 1668A	QAPP Table 2	• Investigate transport of PCBs in the water column • PCB congener analyses essential for analytical sensitivity and distinguishing potential multiple sources of contamination	SPMDs will be used to estimate time-weight-averaged concentrations and bioconcentrations of trace HOCs such as PCBs, PAHs, dioxins, and pesticides. The SPMD data will be used to screen for the presence of certain HOCs in the tributaries and to compare the relative fingerprints of the HOCs in the different locations. Note that the SPMDs will not be used in the HOC sampling methodology validation study.	Assess COPC/COPEC concentrations and transport variation uisng a integrating device for fate and transport modeling; calibrate eutrophication component of model; human and ecological risk assessment. Since the fate and transport model is carbon-based, the calibration of the eutrophication model affects the fate of the chemicals sorbing into the organic fraction.
				b) PAHs	Modified Method 8270 (GC-MS-SIMs)		• Investigate potential transport of COPCs in the water column • Identify and characterize the contaminants • Determine extent of contamination		
				c) Dioxins/furans	Method 1613B		• Investigate transport of dioxins/furans in the water column		
				d) Pesticides (including DDT and metabolites)	Modified Method 8081 (GC-MS-SIMs or GC-ECD)		• Investigate potential transport of COPCs in the water column • Identify and characterize the contaminants • Determine extent of contamination		

Attachment 2

Compilation of Human Health and Ecological Risk-Based Action Levels

	Action Levels - ECOLOGICAL													Action Levels - HUMAN HEALTH			
	Water Quality (marine)			Benthic Organisms (marine sediment)								Fish (marine when available)			Water		Soil	
Chemical	NRWQC - CCC [a]	NYSDEC[b]	NJDEP[c]	ER-L[d]	ER-M[d]	TEL[e]	PEL[e]	Washington State[f]	Canada[g]	EPA EqP Method[h]	AET Method[i]	No Effect Concentration (ERED)[j]	Species	Endpoint	NRWQC - fish consumption[k]	NJDEP[c]	EPA Region 9 HH Soil PRG[l]	NJDEP Soil PRG (residential)
METALS (µg/L water or mg/kg sediment, tissue)																		
Antimony	500 (NOAA, 1999)			2	25										640	4,300	31	31
Arsenic (total)	36			8.2	70	7.2	41.6	57				0.53	Bluegill	Reproduction	0.1	0	0.39	0.4
Arsenic (III)	36																	
Arsenic (V)	13 (OR, 1996)																	
Barium											48					2,000	5,400	5,500
Beryllium											0.36	5.13	Bluegill	Mortality			150	16
Cadmium	8.8			1	10	0.7	4.2	5.1				1	Winter Flounder	Biochemical		10	37	39
Chromium (total)	50			81	370	52.3	160.4	260				0.54	Rainbow trout	Biochemical		3,230	30	
Chromium (VI)	50	54																
Cobalt																	900	1,600
Copper	3.1	3.4	7.9	34	270	18.7	108.2	390				<1.5	Striped mullet (juvenile)	Toxicity		5.6	3,100	3,100
Lead	8.1	8	210	47	218	30.2	112.2	450				0.451	Fathead Minnow	Biochemical		24	400	400
Manganese											480				100	100	1,800	1,600
Mercury (total)	0.9			0.15	0.71	0.1	0.7	0.4				0.09	Spiny Dogfish	Behavior		0.146	23	23
Methylmercury												0.02	Mummichog		0.3 mg/kg		6	
Nickel	8.2	8.2		21	52	15.9	42.8					2.2	Rainbow trout	Biochemical	4,600	3,900	1,600	1,600
Selenium	71										1	0.2	Chinook salmon	Growth	4,200	10	390	390
Silver	1	2.3		1	4	0.7	1.8	6.1				0.044	Bluegill	Growth		164	390	390
Thallium												2.7	Bluegill	Mortality	6.3	6.22	5	16,000
Titanium																	1.0E+05	
Vanadium												0.7	American flagfish	Growth			78	550
Zinc	81	66		150	410	124	271	410				12	Atlantic Salmon	Growth	26,000		23,000	23,000
VOCs (µg/L water or µg/kg sediment, tissue)																		
1,2-Dichloroethylene	224,000 (OR and NH, 1996)															592	69,000	43,000
1,4-Dichlorobenzene		5						3,100		340	35	52,000	Rainbow trout	Development	2,600	3,159	3,400	610,000
1,2,4-Trichlorobenzene	5.4 (Canada, 1999)							810			31	50	Spot	Behavior	940	113	62,000	73,000
Benzene		190								57		1,400	Pacific Herring	Reproduction	51	71	640	3,000
Chlorobenzene		5								820		3,000	Rainbow trout	Physiological	21,000	21,000	150,000	510,000
Ethylbenzene		4.5								1,400	10				29,000	27,900	400,000	7.8E+06
Methyl chloride	6,400 (NH, 1996)																9,100	
SVOCs (µg/L water or µg/kg sediment, tissue)																		
Bis(2-Ethylhexyl)phthalate	3.4 (NH, 1996)					182.2	2,646.5	47,000			1,300				2.2	5.92	35,000	35,000
Biphenyl										1,100	260						3.0E+06	3.1E+06
Butylbenzylphthalate	3.4 (NH, 1996)							4,900		11,000	63	6,450	Bluegill	Mortality	1,900	416	1.2E+07	1.2E+06
Carbazole											970	580	Rainbow trout	Behavior			24,000	24,000
Di-n-Octyl phthalate								58,000			25						2.4E+06	1.2E+06
N-nitroso-di-phenylamine								11,000			28	2,000	Bluegill	Mortality		16.2	99,000	99,000
BUTYLTINS (µg/L water or µg/kg sediment, tissue)																		
Monobutyltin												300	Rainbow trout	Cellular				
Dibutyltin												500	Rainbow trout	Cellular				
Tributyltin	0.01				25							2500	Rainbow trout	Cellular			18,000	
PAHs (µg/L water or µg/kg sediment, tissue)																		
1-Methylphenanthrene											310							
2,3,5-Trimethylnaphthalene											54							
2,6-Dimethylnaphthalene											33							
2-Methylnaphthalene		4.2		70	670	20.2	201				670							
Acenaphthene		6.6		16	500	6.7	88.9			1,300	500	3,500	Bluegill	Mortality	990		3.7E+06	3.4E+06
Acenaphthylene				44	640	5.9	127.9				560							
Anthracene				85	1,100	46.9	245				960				40,000	108,000	2.2E+07	1.7E+07
Fluorene				19	540	21.2	144.4	23,000		540	540	1,800	Rainbow trout	Behavior	5300	1340	2.7E+06	2.3E+06
Naphthalene		16		160	2,100	34.6	390.6			470	2,100	2,300	Mummichog	Biochemical			56,000	2,400,000
Phenanthrene		1.5		240	1,500	86.7	543.5			1,800	1,500	30,000	Rainbow trout	Biochemical				
LMW PAHs				552	3,160	311.7	1,442				5,200							
Benzo[a]pyrene	0.01 (BC, 1998)			430	1,600	88.8	763.2	99,000	500		1,600	14	Yellowspotted rockcod	Growth	0.02	0.031	62	60
Benzo[b]fluoranthene											3,200				0.02	0.031	620	600
Benzo[e]pyrene																		
Benzo[g,h,i]perylene	300 (NH, 1996)							31,000	100		670	27,500	Common carp	Biochemical				
Benzo[k]fluoranthene	300 (NH, 1996)										4,300				0.02	0.031	6,200	6,000

	Action Levels - ECOLOGICAL													Action Levels - HUMAN HEALTH			
	Water Quality (marine)			Benthic Organisms (marine sediment)								Fish (marine when available)			Water		Soil	
Chemical	NRWQC - CCC [a]	NYSDEC[b]	NJDEP[c]	ER-L[d]	ER-M[d]	TEL[e]	PEL[e]	Washington State[f]	Canada[g]	EPA EqP Method[h]	AET Method[i]	No Effect Concentration (ERED)[j]	Species	Endpoint	NRWQC - fish consumption[k]	NJDEP[c]	EPA Region 9 HH Soil PRG[l]	NJDEP Soil PRG (residential)
Chrysene	300 (NH, 1996)			384	2,800	107.8	846				1,400	13,200	Brown bullhead	Lesions/tumor	0.02	0.031	62,000	62,000
Dibenz[a,h]anthracene				63	260	6.2	134.6	12,000			230				0.02	0.031	62	60
Fluoranthene	16 (OR and NH, 1996)			600	5,100	112.8	1,493.5	160,000		6,200	2,500	1250	Rainbow trout	Biochemical	140	393	2.3E+06	2.3E+06
Indeno[1,2,3-c,d]-pyrene	300 (NH, 1996)							34,000			37,000				0.02	0.031	620	600
Perylene																		
Pyrene	300 (NH, 1996)			665	2,600	153	1,398	1,000				30,000	Rainbow trout	Biochemical	4,000	8,970	2.3E+06	1.7E+06
High Molecular Weight PAHs				1,700	9,600	655.3	6,676.1											
PAHs, Total				4,022	44,792	1,684	16,770											
Dibenzothiophene												3,000	Rainbow trout	Behavior				
PCBs - Aroclors (µg/L water or µg/kg sediment, tissue)																		
Aroclor 1016	0.03								100									
Aroclor 1221	0.03																	
Aroclor 1232	0.03																	
Aroclor 1242	0.03											232	Channel catfish	Morphology				
Aroclor 1248	0.03								50									
Aroclor 1254	0.03								63	418		160	Chinook salmon	Growth				
Aroclor 1260	0.03								5			7.6E+06	Fathead Minnow	Reproduction				
PCBs - Congeners (pg/L water or ng/kg sediment, tissue)[pp]																		
PCB 77	40 (BC, 1998)											940,000	Arctic grayling	Biochemical				
PCB 81																		
PCB 105	90 (BC, 1998)																	
PCB 114																		
PCB 118												2.44E+07	Common Carp	Biochemical				
PCB 123																		
PCB 126	0.25 (BC, 1998)											18,000	Common Carp	Biochemical				
PCB 156																		
PCB 157																		
PCB 167																		
PCB 169	60 (BC, 1998)																	
PCB 189																		
PCB 18																		
PCB 28																		
PCB 44																		
PCB 49																		
PCB 52												1.10E+09	Fathead minnow	Reproduction				
PCB 66																		
PCB 101																		
PCB 110																		
PCB 87																		
PCB 128																		
PCB 138																		
PCB 153																		
PCB 170																		
PCB 180												1.21E+09	Fathead minnow	Reproduction				
PCB 183																		
PCB 187																		
PCB 195																		
PCB 206																		
PCB 209																		
Total PCBs	30,000			22,700	180,000	22,000	189,000				1.0E+06	18,100	Dab	Biochemical	64,000	170,000	2.10E+07	200,000
PESTICIDES (µg/L water or µg/kg sediment, tissue)																		
2,4'-DDD											16							
2,4'-DDE											15							
2,4'-DDT											3.9							
4,4'-DDD				2.0	20	1.2	7.8				16	5,000	Brook trout	Growth	0.00031	0.000837	2,400	3,000
4,4'-DDE				2.2	27	2.1	374.2				15	2,400	Lake trout	Behavior	0.00022	0.000591	1,700	2,000
4,4'-DDT	0.001		0.13	1	7	1.2	4.8				3.9	31	Atlantic Salmon	Reproduction	0.00022	0.0006	1,700	2,000
Total DDXs (sum of the six 4,4'- and 2,4'-isomers)				1.58	46.1	3.9	51.7		6.0	1.5	3.0	43,000	Sailfin molly	Physiological				
Aldrin	1.3		1.3								0.44	5,000	Atlantic Salmon	Growth	0.00005	0.00014	29	40
BHC (gamma) Lindane	0.16		0.16			0.32	0.99			3.70		297	Bluegill	Growth	0.019		440	400
BHC (alpha)												30	Guppy	Physiological	0.0026	0.013	90	100
BHC (beta)															0.0091	0.46	320	400
Chlordane	0.004		0.09	0.5	6										0.00081	0.00030	1,600	200

	Action Levels - ECOLOGICAL														Action Levels - HUMAN HEALTH			
	Water Quality (marine)			Benthic Organisms (marine sediment)								Fish (marine when available)			Water		Soil	
Chemical	NRWQC - CCC [a]	NYSDEC[b]	NJDEP[c]	ER-L[d]	ER-M[d]	TEL[e]	PEL[e]	Washington State[f]	Canada[g]	EPA EqP Method[h]	AET Method[i]	No Effect Concentration (ERED)[j]	Species	Endpoint	NRWQC - fish consumption[k]	NJDEP[c]	EPA Region 9 HH Soil PRG[l]	NJDEP Soil PRG (residential)
Dieldrin	0.002		0.710	0.02	8	0.7	4.3					1,200	European plaice	Biochemical	0.000054	0.00014	30	40
Endosulfan (I and II)	0.009	0.001	0.034							5.4		20,000	Australian freshwate	Cellular	89	0.0087	370,000	470,000
Endrin	0.002		0.037	0.02	45					2.15		0	Rainbow Trout	Physiological	0.81	0.0023	18,000	23,000
Heptachlor	0.004		0.053							1.04		4,800	Sheepshead minnow	Cellular	0.000079	0.00020	110	100
Heptachlor epoxide	0.004		0.053						1			4,800	Sheepshead minnow	Cellular	0.000039	0.00011	53	70
Methoxychlor	0.03	0.03							6 (NYDEC, 1994)	19		1,400	Brook trout	Behavior		0.03	3.1E+05	390,000
Toxaphene	0.0002	0.005	0.21						0.1 (NYDEC, 1994)	100		250	Mosquito fish	Physiological	0.0003	0.0002	440	600
2,4,5-T																	6,100	
2,4,5-TP																	490,000	
2,4-D												1,000	Spiny Dogfish	Mortality			690,000	
2,4-DB																	490,000	
DIOXINS/FURANS (pg/L water or ng/kg sediment, tissue)																		
2,3,7,8-TCDD									100 (NYSDEC, 1989)			125	Coho salmon	Growth	0.0051	0.014	39	
1,2,3,7,8-PeCDD												63,800	Common carp	Biochemical				
1,2,3,4,7,8-HxCDD												39	Rainbow Trout	Biochemical				
1,2,3,6,7,8-HxCDD																		
1,2,3,7,8,9-HxCDD																		
1,2,3,4,6,7,8-HPCDD																		
OCDD																		
2,3,7,8-TCDF												2,500	Rainbow trout	Growth				
1,2,3,7,8-PECDF												129,000	Common carp	Biochemical				
2,3,4,7,8-PECDF																		
1,2,3,4,7,8-HXCDF												990	Rainbow Trout	Mortality				
1,2,3,6,7,8-HXCDF																		
2,3,4,6,7,8-HXCDF																		
1,2,3,7,8,9-HXCDF																		
1,2,3,4,6,7,8-HPCDF																		
1,2,3,4,7,8,9-HPCDF																		
OCDF												10,000	Atlantic salmon	Mortality				
WATER QUALITY PARAMETERS																		
Cyanide	1	1	1												220,000	1	11	

(a) Except where noted, values are from the National Recommended Water Quality Criteria for Priority Toxic Pollutants and Nonpriority Pollutants (NRWQC) for seawater; CCC (Criterion Continuous Concentration) is an estimate of the highest concentration of a material in surface water to which an aquatic community can be exposed indefinitely without resulting in an unacceptable effect. Table available at: http://www.epa.gov/waterscience/pc/revcom.pdf

NOAA (National Oceanic and Atmospheric Administration) Screening Quick Reference Table for Inorganics in Water. Proposed Value. September 1999.

OR = Oregon Department of Environmental Quality. 1996. State-wide water quality management plan: beneficial uses, policies, standards, and treatment criteria for Oregon. 178 pp.

NH = New Hampshire Department of Environmental Services. 1996. State of New Hampshire surface water quality regulations. Env-WS 430. 37 pp.

Canada, 1999 = Environment Canada, 1999. Canadian water quality guidelines

BC = British Columbia Ministry of Environment, Lands, and Parks. 1998. British Columbia approved water quality guidelines (Criteria): 1998 Edition. ISBN 0-7726-3680-X. 30 pp.

(b) New York State Department of Environmental Conservation. 1998. Ambient water quality standards and guidance values and groundwater effluent limitations. 124 pp.

(c) New Jersey Department of Environmental Protection Surface Water Quality Standards for human and aquatic endpoints NJAC 7:9B-1.14(c)13. Available online at: http://www.state.nj.us/dep/wmm/sgwqt/2004swqs.pdf

(d) Effects Range - Low (ER-L) and Effects Range Medium (ER-M): Long and Morgan. 1991. The Potential for Biological Effects of Sediment-Sorbed Contaminants Tested in the National Status and Trends Program, NOAA Technical Memorandum NOS OMA 52. Long et al., 1995. Incidence of Adverse Biological Effects within Ranges of Chemical Concentrations in Marine and Estuarine Sediments, *Environmental Management* 19(1): 81-97.

(e) Threshold Effects Level (TEL) and Probable Effects Level (PEL) from MacDonald et al., 1996. Development and evaluation of sediment quality guidelines for Florida coastal waters. *Environ manage* 19:81-97.

(f) Washington State Sediment Quality Chemical Criteria WAC 172-204-320

(g) Miisstere de l'Environment du Quebec et Environnement Canada. 1992. Interim criteria for quality assessment of St. Lawrence River sediment ISBN 0-662-19849-2. St. Lawrence Action Plan.

(h) Equilibrium Partitioning Method. EPA. The incidence and severity of sediment contamination in surface waters of the US. Volume 1: National sediment quality survey. USEPA 823-R-97-006. Office of Science and Technology.

(i) Apparent Effects Threshold Method. Barrick et al.

(j) No observed effect concentration (NOEC) values from ERED (Environmental Residue-Effects Database), available at: http://el.erdc.usace.army.mil/ered/

(k) Values from NRWQC for the protection of human health through fish consumption only. Table available at: http://www.epa.gov/waterscience/pc/revcom.pdf

(l) from Region 9 PRG Table, available at: http://www.epa.gov/region09/waste/sfund/prg/files/04prgtable.pdf and NJDEP Soil Screening Values for Residents. Available at: http://www.nj.gov/dep/srp/regs/srs/proposed/ingestion_dermal_bb.pdf

(m) from USEPA Region 3 Tissue Risk Based Concentration (RBC) Table, available at: http://www.epa.gov/reg3hwmd/risk/human/index.htm

Attachment 3

Quality Assurance Tables Non-CLP Tests

PCB Congeners

USEPA Method 1668, Revision A by HRGC/HRMS

Audits Required	Frequency of Audits	Limits	Action
Initial Precision and Recovery (IPR)	Before analyzing environmental samples and whenever a change is made in the procedure used.	At least four aliquots with diluted labeled compound spiking solution per 1668A, 9.2 and the recovery and RSD criteria in 1668A, Table 6.	An IPR is four aliquots of the diluted (Precision and Recovery Standard) PAR standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed prior to the first time this method is used and any time the method or instrumentation is modified. If the acceptance criteria in not meet, the problem must be solved and the IPR repeated.
Calibration	Prior to analyzing samples	Calibration must follow the requirements given in 1668A, section 10.0.	The calibration requirements must be met before samples are analyzed. The calibration must be repeated if it does not meet acceptance criteria given in 1668A, Section 10.0.
System performance and calibration verification are verified for all native CB and labeled compounds by calibration verification standard and a diluted combined 209 congener solution	At the beginning of each 12-hour shift	All performance criteria given 1668A, section 15.0 and 7.10 Table 5 must be met before samples, blanks, IPRs, and OPRs are analyzed.	All criteria in 1668A must be met before samples are analyzed. Investigate and correct any problems.
MS Resolution	At the beginning and end of each 12 hour shift	Per requirements in 1668A, 15.2. Static resolving power of at least 10,000	The mass spectrometer must be adjusted to meet the require resolution criteria.
Calibration verification (VER)	Beginning and end of every 12 hours of samples run	The theoretical ion abundance ratios for all chlorinated atoms must be within the QC limits in 1668A, Table 8. Peaks for each CB and labeled compound in the VER standard must be present with signal to noise (S/N) of at least 10.	If the control limits are still not met, the analysis must be stopped, the problem corrected, and a new initial calibration check run. Sample analysis cannot begin until the control limits are met.
Retention Times (RT) and GC Resolution	At the beginning of each 12-hour shift	Absolute RTs of labeled Toxics/LOC/window defining congeners ± 15 seconds of RT during calibration. Relative RTs of native CBs and labeled compounds within limits given in Table 2. (see 1668A, 15.4) Must meet resolution and minimum analysis time specifications in 1668A. 6.9.1.1.2 and 6.9.1.1.1.	Gas chromatographic conditions need to be adjusted until the required retention time criteria and resolution are achieved.
Ongoing Precision and Recovery (OPR)	Prior to the analysis of samples from the same batch.	Must meet the OPR limits given in 1668A Table 6.	An OPR is a method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in the method for precision and recovery. If the acceptance criteria in not meet, the problem must be solved and the OPR repeated. If sufficient sample is available any samples associated with the unacceptable OPR should be repeated.
Method Blank	With each sample batch. Analyze immediately before the OPR.	No greater than the minimum detection levels given in 1668A section 9.5.2.	If the method blank exceeds the control limits, corrective actions must be taken, a new method blank must be prepared and analyzed, and all the samples associated with the out of control blank should be re-prepared and reanalyzed.
Spike Samples with Labeled Compound (per 1668A, 9.3)	Each sample must be spiked with diluted labeled compound spiking solution.	Spike recoveries must meet the limits given in 1668A, Table 6.	When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits.

PCB Congeners (Continued)

USEPA Method 1668, Revision A by HRGC/HRMS

Audits Required	Frequency of Audits	Limits	Action
QC Check Sample (QCS) appropriate for each matrix obtained from an independent source. (NIST SRM 1944 is an acceptable certified reference for sediment/solids.)	Prior to analyzing the first batch of samples for the matrix and then at least once a quarter	Must meet the acceptance criteria provided by the supplier of the QC check standard or must be within at least ± 20% of the certified or known values.	A QCS is a sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process. If the criteria are not met the problem must be investigated and corrected before proceeding with additional environmental sample analysis.
For whole water samples the QCS must be a spiked reagent water with a known amount of NIST SRM 1944 (approximately 100 mg of NIST SRM 1944 per liter is recommended)	Prior to analyzing the first batch of whole water samples and at least once a quarter	The PCB recovery for the sum of the certified PCBs (corrected for dilution) must be within 20% of the sum of the certified PCBs congeners in NIST SRM 1944. Recoveries for the individual PCBs 31, 52, 149, and 180 must be also be within 20% of the certified range of values after correction for sample dilution. (This will serve as the QCS for whole water samples)	As a QCS for whole water samples analyze a reagent water samples spiked with a known concentration of NIST SRM 1944. If the data does not meet acceptance criteria then optimize the sample extraction/ cleanup procedure and re-prepare and reanalyze the QCS. Report the recoveries in the case narrative. Report any problems to the Malcolm Pirnie representative.
Duplicate	With each batch of up to 20 samples	Must agree to within ± 20% of the mean (applicable to concentrations 10 times the detection limits)	If the limits are not met; verify satisfactory instrument performance; verify that no error was made while weighing the sample and reagents; review the analytical procedure with the laboratory personnel; note the findings in the case narrative.
Field Duplicate	Typically with each batch of samples	For aqueous samples must agree to within ± 40 % of the mean (applicable to concentrations 10 times the detection limits. For sediment samples must agree to within ± 50 % of the mean (applicable to concentrations 10 times the detection limits.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Dioxins/Furans

USEPA Method 1613B Tetra through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS., October 1994

Audits Required	Frequency of Audits	Limits	Action
Initial Precision and Recovery (IPR)	Before analyzing environmental samples and whenever a change is made in the procedure used.	Four aliquots with diluted labeled compound spiking solution per 1613, 9.2 and the criteria in 1613, Table 6.	An IPR is four aliquots of the diluted (Precision and Recovery Standard) PAR standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed prior to the first time this method is used and any time the method or instrumentation is modified. If the acceptance criteria in not meet, the problem must be solved and the IPR repeated.
Calibration System performance and calibration verification are verified for all analytes and labeled compounds by calibration verification standard and isomer specificity test standards..	Prior to analyzing samples At the beginning of each 12-hour shift	Calibration must follow the requirements given in 1613, section 10.0. All performance criteria given 1613, Tables 4 and 5 must be met before samples, blanks, IPRs, and OPRs are analyzed.	The calibration requirements must be met before samples are analyzed. The calibration must be repeated if it does not meet acceptance criteria given in 1613, section 10.0.
Mass Spectrometer (MS) Resolution	At the beginning and end of each 12 hour shift	Per requirements in 1613, 15.2. Static resolving power of at least 10,000	The mass spectrometer must be adjusted to meet the require resolution criteria.
Calibration verification (VER)	Beginning and end of every 12 hours of samples run	The m/z abundance ratios shall be within the limits in 1613, Table 9. Peaks for each analyte and labeled compound in the VER standard must be present with S/N of at least 10.	If the control limits are still not met, the analysis must be stopped, the problem corrected, and a new initial calibration check run. Sample analysis cannot begin until the control limits are met.
Retention Times (RT) and GC Resolution	At the beginning of each 12-hour shift	Absolute RTs of GCMS internal standards ± 15 seconds of RT during calibration. Relative RTs within limits given in Table 2. (see 1613, 15.4)	Gas chromatographic conditions need to be adjusted until the required retention time criteria and resolution are achieved.
Ongoing Precision and Recovery (OPR)	Prior to the analysis of samples from the same batch.	Must meet the OPR limits given in 1613 Table 6.	An OPR is a method blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in the method for precision and recovery. If the acceptance criteria in not meet, the problem must be solved and the OPR repeated. If sufficient sample is available any samples associated with the unacceptable OPR should be repeated.
Method Blank	With each sample batch. Analyze immediately before the OPR.	No greater than the minimum detection levels given in 1613. (see 1613, 9.5)	If the method blank exceeds the control limits, corrective actions must be taken, a new method blank must be prepared and analyzed, and all the samples associated with the out of control blank should be re-prepared and reanalyzed.
Spike Samples with Labeled Compound (per 1613, 9.3)	Each sample must be spiked with diluted labeled compound spiking solution.	Spike recoveries must meet the limits given in 1613, Table 7 and or 7a.	When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits.
QC Check Sample obtained from and independent source	Analyze at least once a quarter	Most meet the acceptance criteria provided by the supplier of the QC check standard or must be within at least ± 20% of the certified or known values.	A QCS is a sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process. If the criteria are not met the problem must be investigated and corrected before proceeding with additional environmental sample analysis.
Duplicate	With each batch of up to 20 samples	Must agree to within ± 20% of the mean (applicable to concentrations 10 times the detection limits)	If the limits are not met; verify satisfactory instrument performance; verify that no error was made while weighing the sample and reagents; review the analytical procedure with the laboratory personnel; note the findings in the case narrative.
Field Duplicate	Typically with each batch of samples	Must agree to within ± 40 % of the mean (applicable to concentrations 10 times the detection limits	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

$Dioxins_{TEQ}$ and PCB_{TEQ} through Immunoassay

A modified version of EPA SW-846-4025, Screening for Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans (PCDD/Fs) by Immunoassay. The modifications to 4025, with cleanup based upon portions of 8290, allow for screening for $Dioxin_{TEQ}$ and PCB_{TEQ}. The method is based upon Cape-Technologies DF-1 Dioxin/Furan Immunoassay Kit plus the PCB1 Insert (IN-PCB1). See QAPP Attachments 7 and 8 for more information.

Audits Required	Frequency of Audits	Limits	Action
Initial Site/Matrix Split Sample Correlation	Before full scale implementation of the technique for the project	The Immunoassay results will be correlated with Dioxin data provided by an approved lab by HRGC-HRMS (1613 for Dioxins/Furans and 1668A for PCBs) on at least 20 split samples.	Initial site and matrix specific split sample correlation studies will be conducted on a set of approximately 20 samples with dioxin including sample with results blow the reporting limits and at least an order of magnitude above the reporting limit. This will be completed prior to full scale implementation of the technique for the project. The Immunoassay results will be correlated with dioxin data provided by an approved CLA lab by HRGC-HRMS. From this data calibration adjustment factors will be determined. Since the HRGC-HRMS method employs internal standards to correct for sample preparation efficiencies, for this study the HRGC-HRMS data will be considered as having no bias. For this study a correlation coefficient of 0.80 would be considered to be suitable. If these criteria can not be achieve the problem will be documented.
On going split sample conformation	At least 10 percent of the samples for the first 200 samples. After that the frequency will be re-evaluated.	Document – These data will provide confirmation of the method correlation. The ideal RPD between methods would be <25%.	Split sample analyses for dioxins on 10% of the first 200 samples by HRGC-HRMS will be used for on-going confirmation and possible further optimization of the calibration adjustment factors. After the first 200 samples the need and frequency for confirmation samples will be reevaluated. These data will provide confirmation of the method correlation.
Method Detection Limit	Prior to the lab analyzing environmental samples and whenever a change is made in the method which could change the detection limit	Sufficient to meet requirements for screening reporting Limit of 20 ppt TEQ	Prior to the lab analyzing environmental samples and whenever a change is made in the method, which would alter the detection limit. The MDL should be low enough to support the screening reporting limit.
Method Blank	For each matrix, at least one per batch of 20 samples	< RL	If the method blank is above the detection limit investigate the source of the problem prior to analyzing samples.
Matrix Spikes	For each matrix, at least one per batch of 20 samples	Recovery greater than 40%	Record the MS recovery for the matrix and report in the case narrative.
Standard Reference Material (SRM)	Initially at the beginning of the project and than at least semi- annually	± 35% of expected	If the SRM result does not meet expected values the cause of the problem should be investigated prior to analyzing samples.
Fortified Method Blank (FMB)	At least one per 20 samples	± 30% of expected	If the result falls outside the control limits, another FMB should be analyzed. If this is also outside the limits the problem must be investigated further and documented.
Duplicate	Weekly when samples are tested	RPD < 50% evaluated for analytes >5 times the MDL.	If the limits are exceeded for the duplicate, record in the case narrative.

Polynuclear Aromatic Hydrocarbons

Modified USEPA Method SW-846 8270, employing GC/MS-SIMs.

Audits Required	Frequency of Audits	Limits	Action
Initial calibration	Prior to analyzing samples.	A five point curve (minimum) for each compound of interest covering the range of the sample being analyzed.	The initial calibration requirements must be met before any samples are analyzed. If any continuing calibration does not meet the required criteria, a new initial calibration sequence must be run. The initial calibration sequence must consist of a minimum of at least five (5) standard concentrations. If the calibration curve does not meet the required limit, fresh standards must be analyzed and a new standard curve generated. If a sample concentration is at or exceeds the highest calibration standard, the sample should be reanalyzed using a dilution.
Calibration verification (Using mid-point QC check)	Beginning and end of every 12 hours of samples run	Standards must fall within the absolute retention time windows. Results must be within ± 20% of the response calculated using the initial calibration.	A mid-point continuing calibration QC check standard must be run at the beginning and end of every 12 hours of sample analysis per instrument. If standards do fall within the absolute retention windows the GC retention times should be corrected prior to analyzing samples. Results must be within ± 20% of the response calculated using the initial calibration. If control limits are not met, corrective actions must be taken, and a new continuing calibration check sample run. If the control limits are still not met, the analysis must be stopped, the problem corrected, and a new initial calibration check run. Sample analysis cannot begin until the control limits are met. To validate positive data, the continuing calibration check must also be acceptable at the end of every 12 hour period during which samples are analyzed. Samples must be reanalyzed if the ending continuing calibration check control limits are not met.
Method Blanks	With each batch of up to 20 samples	< 3 x detection limit	All sample results must be associated with an acceptable method blank which was extracted within the same extraction time, batch, and matrix type as the samples. A method blank is required between a calibration run and the first sample run. The method blank and the samples must be analyzed on the same instrument. If the method blank exceeds the control limits, corrective actions must be taken, including documenting and justifying the exceedence(s), reanalyzing the method blank, and/or reprocessing the entire batch.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	With each batch of up to 20 samples	Percent Recovery (%R) – 40-120% Relative Percent Difference (RPD) - ≤30% Target spike must be >5x background concentration to be appropriate for data quality assessment.	The MS fortification solutions are to contain all the unlabeled analytes at concentrations corresponding to the calibration mid-point. The MD must have a recovery of at least 40-120%. The results obtained from the MD and MSD samples should agree within 30 percent relative difference. If the limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Laboratory Control Sample (LCS)	With each analytical batch of up to 20 samples	%R – 40-120%.	An LCS must be analyzed with each analytical batch of up to 20 samples. The LCS consists of an aliquot of a clean control matrix similar to the sample matrix and of the same weight and volume. The LCS is spiked with the same analytes at the same concentrations as the MS. Recovery must be at least 40-120%. When the results of the MS indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
Surrogate Recoveries	With each analysis, before sample extraction, spike each sample and standard with pesticide surrogates.	%R – 40-120%	With each analysis, before sample extraction, spike each sample and standard with one or two herbicide surrogates. Develop statistically determined QC chart limits with recovery limits not more than 50-120%. QC check samples should be re-extracted and re-analyzed if surrogate recovery does not meet control limits.
QC Check Sample for Whole Water Samples. Analyze a reagent water samples spiked with a known concentration of NIST SRM 1944	Prior to analyzing the first batch of whole water samples and at least once per quarter during which samples are analyzed.	The recoveries (After correction for dilution) for the certified concentrations of Phenanthrene, Benzo(a) pyrene, Chrysene and Pyrene must be within 30% of the corrected certified range of values	As a QC check for whole water samples analyze a reagent water samples spiked with a known concentration of NIST SRM 1944. If the data does not meet acceptance criteria optimize the sample preparation procedure. Record all the recoveries in the case narrative. Report problems to the Malcolm Pirnie representative.
Duplicate Samples	With each batch of up to 20 samples	≤ 30 % RPD or Diff < detection limit Analyte concentration must be >5x MDL to be appropriate for data quality assessment.	The results of the laboratory duplicates should agree within ±30 RPD (difference expressed as percentage of the mean). If the limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Field Duplicate	Per batch of 20 samples	≤ 50 % RPD or Diff < detection limit	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.
Rinsate Blank	Not to exceed one Rinsate per day of sampling, but at least one weekly	< detection limits	Any problems with the rinsate blanks will be addressed by the data validator, not the laboratory.

Pesticides

Modified USEPA Method 8081, employing GC/MS-SIMs and GC/ECD

Audits Required	Frequency of Audits	Limits	Action
Initial calibration	Prior to analyzing samples.	A five point curve (minimum) for each compound of interest covering the range of the sample being analyzed.	The initial calibration requirements must be met before any samples are analyzed. If any continuing calibration does not meet the required criteria, a new initial calibration sequence must be run. The initial calibration sequence must consist of a minimum of at least five (5) standard concentrations. If the calibration curve does not meet the required limit, fresh standards must be analyzed and a new standard curve generated. If a sample concentration is at or exceeds the highest calibration standard, the sample should be reanalyzed using a dilution.
Calibration verification (Using mid-point QC check)	Beginning and end of every 12 hours of samples run	Standards must fall within the absolute retention time windows. Results must be within ± 20% of the response calculated using the initial calibration.	A mid-point continuing calibration QC check standard must be run at the beginning and end of every 12 hours of sample analysis per instrument. If standards do fall within the absolute retention windows the GC retention times should be corrected prior to analyzing samples. Results must be within ± 20% of the response calculated using the initial calibration. If control limits are not met, corrective actions must be taken, and a new continuing calibration check sample run. If the control limits are still not met, the analysis must be stopped, the problem corrected, and a new initial calibration check run. Sample analysis cannot begin until the control limits are met. To validate positive data, the continuing calibration check must also be acceptable at the end of every 12 hour period during which samples are analyzed. Samples must be reanalyzed if the ending continuing calibration check control limits are not met.
Method Blanks	With each batch of up to 20 samples	< 3 x detection limit	All sample results must be associated with an acceptable method blank which was extracted within the same extraction time, batch, and matrix type as the samples. A method blank is required between a calibration run and the first sample run. The method blank and the samples must be analyzed on the same instrument. If the method blank exceeds the control limits, corrective actions must be taken, including documenting and justifying the exceedence(s), reanalyzing the method blank, and/or reprocessing the entire batch.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	With each batch of up to 20 samples	Percent Recovery (%R) – 40-120% Relative Percent Difference (RPD) - ≤30% Target spike must be >5x background concentration to be appropriate for data quality assessment.	If the limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Laboratory Control Sample (LCS)	With each analytical batch of up to 20 samples	%R - 40-120%.	An LCS must be analyzed with each analytical batch of up to 20 samples. The LCS consists of an aliquot of a clean control matrix similar to the sample matrix and of the same weight and volume. The LCS is spiked with the same analytes at the same concentrations as the MS. When the results of the MS indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.
Surrogate Recoveries	With each analysis, before sample extraction, spike each sample and standard with one or two herbicide surrogates.	%R – 40-120%	With each analysis, before sample extraction, spike each sample and standard with one or two herbicide surrogates. Develop statistically determined QC chart limits with recovery limits not more than 50-120%. QC check samples should be re-extracted and re-analyzed if surrogate recovery does not meet control limits.
QC Check Sample for Whole Water Samples. Analyze a reagent water samples spiked with a known concentration of NIST SRM 1944	Prior to analyzing the first batch of whole water samples and at least once per quarter during which samples are analyzed.	The recovery for the certified concentrations (after correction for dilution) of 4,4'DDT must be at within 30% of the corrected certified range of the value	
Duplicate Samples	With each batch of up to 20 samples	≤ 30 % RPD or Diff < detection limit Analyte concentration must be >5x MDL to be appropriate for data quality assessment.	If the limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Field Duplicate	Per batch of 20 samples	≤ 50 % RPD or Diff < detection limit	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.
Rinsate Blank	Not to exceed one Rinsate per day of sampling, but at least one weekly	< detection limits	Any problems with the rinsate blanks will be addressed by the data validator, not the laboratory.

Chlorinated Herbicides

USEPA SW-846 Method 8151A, Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzlation Derivatization plus any additional cleanup required for sediment samples.

Audits Required	Frequency of Audits	Limits	Action
Initial calibration	Prior to analyzing samples.	A five point curve for each compound of interest covering the range of the sample being analyzed and at least down to the RL.	The initial calibration requirements must be met before any samples are analyzed. If any continuing calibration does not meet the required criteria, a new initial calibration sequence must be run. The initial calibration sequence must consist of a minimum of at least five (5) standard concentrations. If the calibration curve does not meet the required limit, fresh standards must be prepared and a new standard curve generated. If a sample concentration is at or exceeds the highest calibration standard, the sample should be reanalyzed using a dilution.
Calibration verification (Using mid-point QC check)	Beginning and end of every 12 hours of samples run	Standards must fall within the absolute retention time windows. Results must be within ± 15% of the response calculated using the initial calibration.	A mid-point continuing calibration QC check standard must be run at the beginning and end of every 12 hours of sample analysis per instrument. If standards do fall within the absolute retention windows the GC retention times should be corrected prior to analyzing samples. Results must be within ± 15% of the response calculated using the initial calibration. If control limits are not met, corrective actions must be taken, and a new continuing calibration check sample run. If the control limits are still not met, the analysis must be stopped, the problem corrected, and a new initial calibration check run. Sample analysis cannot begin until the control limits are met. To validate positive data, the continuing calibration check must also be acceptable at the end of every 12 hour period during which samples are analyzed. Samples must be reanalyzed if the ending continuing calibration check control limits are not met.
Method Blanks	Beginning of every 12 hours of the sample run and per batch	<RL	All sample results must be associated with an acceptable method blank which was extracted within the same extraction time, batch, and matrix type as the samples. A method blank is required between a calibration run and the first sample run. Therefore, the same method blank extract may be analyzed more than once if the number of samples within a batch requires more than 12 hours of analyses. The method blank and the sample must be analyzed on the same instrument. If the method blank exceeds the control limits, corrective actions must be taken, a new method blank must be prepared and analyzed. Note action taken in the case narrative.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	With each batch of up to 20 samples	Percent Recovery (%R) - 40-120%. Relative Percent Difference (RPD) - ≤20%	The MS fortification solutions are to contain all the unlabeled analytes at concentrations corresponding to the calibration mid-point. The MD must have a recovery of at least 40-120%. The results obtained from the MD and MSD samples should agree within 20 percent relative difference. If the limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Laboratory Control Sample (LCS)	With each analytical batch of up to 20 samples	%R - 70-130%.	An LCS must be analyzed with each analytical batch of up to 20 samples. The LCS consists of an aliquot of a clean control matrix similar to the sample matrix and of the same weight and volume. The LCS is spiked with the same analytes at the same concentrations as the MS. When the results of the MS indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If LCS results are outside limits the problem needs to be investigated and if necessary the batch of samples reanalyzed. Note findings in case narrative.
Surrogate Recoveries	With each analysis, before sample extraction, spike each sample and standard with one or two herbicide surrogates.	Use statistically determined QC chart limits. %R - 50-120%	With each analysis, before sample extraction, spike each sample and standard with one or two herbicide surrogates. Develop statistically determined QC chart limits with recovery limits not more than 50-120%. QC check samples should be re-analyzed if surrogate recovery does not meet control limits. Note findings in case narrative.
Duplicate Samples	With each batch of up to 20 samples	≤ 35 % RPD; evaluated for analytes > 5 times MDL.	If the limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Field Duplicate	Per batch of 20 samples	≤ 50 % RPD; evaluated for analytes > 5 times MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.
Rinsate Blank	Not to exceed one Rinsate per day of sampling, but at least one weekly	< RL	Any problems with the Rinsate blanks will be addressed by the data validator, not the laboratory.

Butyl tin compounds

In the absence of USEPA methods for analysis of butyl tin compounds, the laboratory will follow their on SOPs employing GC

Audits Required	Frequency of Audits	Limits	Action
Initial calibration (ICAL)	Initially prior to analyzing samples	A five-point curve (minimum) for each compound of interest covering the range of the sample being analyzed. ≤25% Relative Standard Deviation (RSD) $r^2 \geq 0.995$	The initial calibration requirements must be met before any samples are analyzed. If any continuing calibration does not meet the required criteria, a new initial calibration sequence must be run. The initial calibration sequence must consist of a minimum of five (5) standard concentrations. If the calibration curve does not meet the required limit, standards must be reanalyzed and a new standard curve generated. If a sample concentration is at or exceeds the highest calibration standard, the sample should be diluted and reanalyzed.
Initial Calibration Check	Once with each initial calibration	Percent Difference from ICAL ≤15%	
Calibration Verification (Using mid-point QC check)	At the beginning and end of every 12 hours of samples run.	Standards must fall within the absolute retention time windows. Results must be within ± 25% of the response calculated	A mid-point continuing calibration QC check standard must be run at the beginning and end of every 12 hours of sample analysis per instrument. If standards do not fall within the absolute retention windows the GC retention times should be corrected prior to analyzing samples. If control limits are not met, corrective actions must be taken, and a new continuing calibration check sample run. If the control limits are still not met, the analysis must be stopped, the problem corrected, and a new initial calibration sequence must be run. Sample analysis cannot begin until the control limits are met. To validate positive data, the continuing calibration check must also be acceptable at the end of every 12 hour period during which samples are analyzed. Samples must be reanalyzed if the ending continuing calibration check control limits are not met.
Internal Standards (IS)	Every sample prior to analysis	Area within 50-200% and retention time within 0.5 min of IS in associated calibration standard.	
Method Blanks	With each batch of up to 20 field samples	< RL or analyte concentrations in associated samples > 10 times blank concentrations	All sample results must be associated with an acceptable method blank which was extracted within the same extraction time, batch, and matrix type as the samples. A method blank is required between a calibration run and the first sample run. The method blank and the samples must be analyzed on the same instrument. If the method blank exceeds the control limits, corrective actions must be taken, including investigating and justifying the reason the control limits were exceeded, reanalyzing the method blank, and/or reprocessing the entire batch; note the findings in the case narrative.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	With each batch of up to 20 field samples	Percent Recovery (%R): Water: 10-45% Relative Percent Difference (RPD) - ≤50% Target spike must be >5 times background concentration to be appropriate for data quality assessment.	A matrix spike and matrix spike duplicate (MS/MSD) must be analyzed with each analytical batch of up to 20 samples. The MS fortification solutions are to contain all the unlabeled target analytes at concentrations corresponding to the calibration mid-point. If the quality control limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Laboratory Control Sample (LCS)	With each analytical batch of up to 20 field samples	%R: 50-150%.	An LCS must be analyzed with each analytical batch of up to 20 samples. The LCS consists of an aliquot of a clean control matrix similar to the sample matrix and of the same weight and volume. The LCS is spiked with the same analytes at the same concentrations as the MS. When the results of the MS indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the QC limits are not met, verify satisfactory instrument performance and review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Surrogate Recoveries	With each analysis, before sample extraction, spike each sample with surrogates.	%R: 30-120%	With each analysis, before sample extraction, spike each sample with surrogates. Develop statistically determined QC chart limits with recovery limits defined in QC Requirements table (item 3). QC check samples should be re-extracted and re-analyzed if surrogate recovery does not meet control limits.
Laboratory Duplicate Samples	With each batch of up to 20 samples	≤ 30 % RPD or % Diff < RL Analyte concentration must be >5 times MDL to be appropriate for data quality assessment.	A laboratory duplicate must be analyzed with each analytical batch of up to 20 samples. Duplicate samples are aliquots of similar mass or volume taken from the same sample container and carried through the entire preparation and analytical procedure. If the QC limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Field Duplicate	Per 20 samples	≤50% RPD; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.
Rinsate Blank	Not to exceed one Rinsate per day of sampling, but at least one weekly	< RL	Results of Rinsate blank analyses that exceed recommended limits for analytes of interest will be addressed by the data validator, not the laboratory.

Total Petroleum Hydrocarbons

New Jersey DEP Method Using GC/FID

Audits Required	Frequency of Audits	Limits	Action
Initial calibration (ICAL)	Prior to analyzing samples	A five-point curve using the external standard techniques described in USEPA SW-848 8015B using a representative standard such as No. 2 Diesel fuel should be used to calibrate the instrument.	The calibration requirements must be met before any samples are analyzed. The initial calibration sequence must consist of a minimum of five (5) standard concentrations. If a sample concentration is at or exceeds the highest calibration standard, the sample should be diluted and reanalyzed or a new calibration point added above the concentration of the sample.
Method Blanks	With each batch of up to 20 field samples	< RL or analyte concentrations in associated samples > 10 times blank concentrations	All sample results must be associated with an acceptable method blank which was extracted within the same extraction time, batch, and matrix type as the samples. A method blank is required between a calibration run and the first sample run. The method blank and the samples must be analyzed on the same instrument. If the method blank exceeds the control limits, corrective actions must be taken, including investigating and justifying the reason the control limits were exceeded, reanalyzing the method blank, and/or reprocessing the entire batch; note the findings in the case narrative.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	With each batch of up to 20 field samples	Percent Recovery (%R): 40-140% In water Relative Percent Difference (RPD) - ≤25% In sediment Relative Percent Difference (RPD) - ≤35%	A matrix spike and matrix spike duplicate (MS/MSD) must be analyzed with each analytical batch of up to 20 samples. If the quality control limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Laboratory Control Sample (LCS)	With each analytical batch of up to 20 field samples	%R: 40-140%.	An LCS must be analyzed with each analytical batch of up to 20 samples. The LCS consists of an aliquot of a clean control matrix similar to the sample matrix and of the same weight and volume. The LCS is spiked with the same analytes at the same concentrations as the MS. When the results of the MS indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If the QC limits are not met, verify satisfactory instrument performance and review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Surrogate Recoveries	With each analysis, before sample extraction, spike each sample with surrogates	%R: 40-150%	With each analysis, before sample extraction, spike each sample with surrogates. Develop statistically determined QC chart limits with recovery limits defined in QC Requirements table (item 3). QC check samples should be re-extracted and re-analyzed if surrogate recovery does not meet control limits.
Laboratory Duplicate Samples	With each batch of up to 20 samples	≤ 30 % RPD or % Diff < RL Analyte concentration must be >10 times MDL to be appropriate for data quality assessment.	A laboratory duplicate must be analyzed with each analytical batch of up to 20 samples. Duplicate samples are aliquots of similar mass or volume taken from the same sample container and carried through the entire preparation and analytical procedure. If the QC limits are not met: verify satisfactory instrument performance; if possible, verify that no error was made while weighing the sample portions; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Field Duplicate	With each batch of samples	RPD < 50%; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Total Organic Carbon (TOC) and Dissolved Organic Carbon (DOC)

EPA SW-846-9060, Total Organic Carbon. To measure Dissolved Organic Carbon (DOC) filter the sample through a 0.45 µm filter in accordance with USEPA Method 415.3. Samples will be analyzed according to the method "Determination of Total Organic Carbon in Sediment," July 27, 1988, by L. Kahn of USEPA.

Audits Required	Frequency of Audits	Limits	Action
Instrument Calibration	Daily, prior to sample analysis	90 - 110 % R	Instrument calibration is to be performed daily prior to analysis according to the instrument manufacturer's instructions. The initial calibration sequence shall consist of a minimum of at least four (4) standards; one (1) blank and three (3) standards in graduated amounts which bracket the expected range of analysis. One (1) calibration standard must be near the instrument's detection limit. If the initial calibration curve does not meet the required limit, the curve must be rerun. The control limit must be met prior to sample analysis. The true values and the source of the verification standards and identification information must be supplied. For samples which exceed the calibration range, a new calibration curve must be prepared which encompasses a higher concentration range. The laboratory must demonstrate that the calibration curve is linear throughout the extended range.
Preparation Blank	1 per sample batch	< RL	The preparation blank must follow the exact analytical procedure as the field samples. All positive sample results must be associated with an acceptable blank. If the preparation blank exceeds the control limits, the instrument should be recalibrated and the preparation blank re-prepared and reanalyzed. The blank acceptance criteria must be met prior to sample analysis.
Mid-range Continuing Calibration Verification (CCV)	Immediately after the instrument calibration and 1 per 10 samples	80 - 120 % R	If the mid-range continuing calibration verification control limits are not met, the analysis must be stopped and the problem corrected. The instrument will then be recalibrated, the calibration verified, and all the samples since the last compliant mid-range calibration verification will be reanalyzed. All positive detections must be associated with an acceptable calibration. The initial calibration verification standard must be prepared from a source other than that used to prepare the calibration standards.
Continuing Calibration Blank	Immediately after the mid-range CCV and 1 per 10 samples	≤ MDL	All positive sample results must be associated with an acceptable CCB. If the CCB exceeds the control limits, the analysis must be stopped and the problem corrected. The preceding ten (10) samples analyzed since the last compliant CCB must also be reanalyzed.
Duplicate Analysis	Every sample	RPD ≤ 20%	Duplicate analyses which exceed the control limits must be reported in the case Narrative.
Quadruplicate Analysis (Sediments only)	1 per sample batch of sediment samples and for each sample with an aliquot < 50 mg	< 3 standard deviations	For sediment samples take one sample per batch of 20 or less and analyze in quadruplicate and for each sediment sample with an aliquot < 50 mg. calculate the standard deviation. If the sample being run in quadruplicate exceeds the control limits, the analysis must be stopped and the problem identified. All the samples in that batch, as well as the quadruplicate sample, must be rerun. The laboratory should report both determinations.
Field Duplicate	With each batch of 20 samples	RPD ≤ 50%; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Particulate Organic Carbon (POC)

USEPA Method 440.0 or modified Lloyd Kahn

Audits Required	Frequency of Audits	Limits	Action
Preparation Blank	1 per sample batch [a]	<RL	The preparation blank must follow the exact analytical procedure as the field samples. All positive sample results must be associated with an acceptable blank. If the preparation blank exceeds the control limit, then the instrument should be recalibrated and the preparation blank re-prepared and re-analyzed. The blank acceptance criteria must be met prior to sample analysis.
Laboratory Duplicate [b]	1 per sample batch	RPD 20 % evaluated for analytes >5 times the MDL.	Duplicate sample analyses, which exceed the control limits, must be reported in the case narrative.
Laboratory Fortified Blank	1 per sample batch	± 25%	Known quantities of the method analytes are added to an aliquot of reagent water, or other blank matrices, in the laboratory. This blank is analyzed exactly like a sample, and its purpose is to determine whether the method is in control, and whether the laboratory is capable of making accurate and precise measurements (see USEPA Method 440.0, Part 3.14)
Laboratory Fortified Sample Matrix	1 per sample batch	± 25%	Known quantities of the method analytes are added to an aliquot of an environmental sample in the laboratory. This laboratory fortified sample matrix is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the laboratory fortified sample matrix corrected for background concentrations (see USEPA Method 440.0, Part 3.15).

Trace Mercury and Methyl Mercury

USEPA Method 1630 Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and CVAFS (EPA-821-R-01-020) with modifications including sample preparation steps for the extraction of sediment
Appendix to USEPA Method 1631, Total Mercury in Tissue, Sludge, Sediment and Soil by Acid Digestion and BrCl Oxidation, and USEPA Method 1631, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry

Audits Required	Frequency of Audits	Limits	Action
Matrix Spike (MS) Matrix Spike Duplicate (MSD)	5% of field samples from site. 5 % of samples from site.	Total Mercury %R - 75-125% Methyl Mercury %R - 65%-135% Total Mercury %R - 75-125% Methyl Mercury %R - 65%-135% RPD - ≤ 25% for water, RPD - ≤ 30% for sediment	If a MS exceed the recovery limits of 75-125% for total mercury or 65%-135% for methyl mercury, verify satisfactory instrument performance. If the RPD exceeds 25%, verify that no error was made preparing the spikes, review the analytical procedure with the performing laboratory personnel and note the findings and correction actions in the case narrative.
Method Blanks (Matrix for the blanks must match the sample matrix for the batch of samples)	Three method blanks should accompany each analytical batch.	< RL	The method blanks are reagent blanks prepared and analyzed exactly as if they were samples. Three method blanks should accompany each analytical batch. If above the limits, the lab should investigate the source of contamination. (Method blanks could be higher for solid sample matrix and sediments) If the method blank exceeds the control limits, corrective actions must be taken, a new method blank must be prepared and analyzed. Note finding in case narrative.
Trip Blank	One with each set of samples and analyze immediately before samples	< RL	If contamination is detected the sampling coordinator must notified. Note in the case narrative
Rinsate Blank	Before sampling using sampler. Not to exceed one Rinsate per day of sampling, but at least weekly	< RL	Any problems with the Rinsate blanks will be addressed by the data validator, not the laboratory.
Ongoing Precision and Recovery (OPR)	At the beginning of each analytical batch and at the end of each 12-hour shift.	The lab should plot OPR data on control charts and develop statement of lab quality for the analysis per EPA 1630 or 1631, Section 9.5 and table 2. Recovery for Total Mercury: 80-120%; Methyl Mercury: 75-125%. Is acceptable.	The OPR is a calibration check standard. If the OPR standard exceed the criteria given in EPA method, the associated results are suspect. The problem needs to be investigated and correction action taken.
Quality Control Standard (QCS)	Analyzed in the middle of each sample batch.	Compare to expected value for the QCS. Difference between the expected value and the results should not exceed ± 25% for Total Mercury and + 35% for Methyl Mercury..	The QCS should be prepared from an independent source than used for standards either as a Laboraotry Fortified Blank (LFB) or a Certified Reference Material (CRM) that is prepared and analyzed with the samples. The difference between the expected value for the QCS and the result should not exceed be greater than ±25% for Total Mercury or ±35% for Methyll Mercury . If the QCS exceeds the limits, investigate and correct the problem.
Field Duplicate	With each batch of samples	RPD ≤ 50%; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Arsenic Species

USEPA Method 1632 Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry, Revision A with modified for total Asenic and for Arsenic species in sediment sample.

Audits Required	Frequency of Audits	Limits	Action
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	On 5% of the samples from the site or at least one MS/MSD for each sample set from the site, whichever is more frequent.	%R - 60-140% %RPD ≤20% for water & ≤30% for sediment; evaluated for analytes >5 times the MDL.	If the MS recovery limits are not met verify satisfactory instrument performance or if for the MSD the RPD exceeds 20% for water or 30% for sediment, verify that no error was made preparing the spikes; review the analytical procedure with the performing laboratory personnel; note the findings in the case narrative.
Method Blanks	For each analytical batch.	< RL	Matrix for the blanks must match the sample matrix for the batch of samples. If above the RL for the matrix, the lab should halt the analysis and investigate the source of contamination. A fresh method blank should be reanalyzed. Note in the case narrative
Rinsate Blank	Before sampling using a sampler. Not to exceed one Rinsate per day of sampling, but at least weekly	< RL	Any problems with the Rinsate blanks will be addressed by the data validator, not the laboratory.
Ongoing Precision and Recovery	At the beginning of each analytical batch and at the end of each 12-hour shift.	For each matrix calculate %R and standard deviation of recovery (SR) per 1632 Section 9.6.5. %R - 60-140%	The OPR is a laboratory fortified method blank. If they exceed the limits any associated results maybe suspect. The problem needs to be investigated and corrective action taken.
Quality Control Standard	Per batch of samples.	%R – 75-125% for water, 65-135% for sediment	The QCS is used to verify instrument calibration; The QCS should be prepared from an independent source other than used for standards. If the QCS exceeds recovery limits, investigate and recalibrate the instrument if necessary.
Field Duplicates	Per sample batch	RPD ≤ 50%; evaluated for analytes >5 times the MDL	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Hexavalent Chromium

EPA Method SW-846-7199, Determination of Hexavalent Chromium in Drinking Water, Groundwater and Industrial Wastewater Effluents by Ion Chromatography with modifications including sample preparation procedure for sediment such as SW 846 3060A.

Audits Required	Frequency of Audits	Limits	Action
Method Blank	Per matrix, at least one per batch of 20 samples of less	<RL	If the method blank is above the detection limit investigate the problem prior to analyzing samples.
Laboratory Duplicates	Per matrix, at least one per 10 samples	Compare the duplicate difference to statistically developed control chart limit with minimum limits of 20%.	A duplicate sample or a duplicate MS sample should be analyzed every ten samples. Compare the duplicate difference to statistically developed control chart limit with minimum limits of 20%. Duplicate analyses which exceed the control limits must be reported in the case narrative.
Matrix Spike (MS)	Per matrix, at least one per batch of 20 samples	For aqueous %R – 85-115% For sediment %R – 75-125%	When ever a new matrix is analyzed MS samples should be analyzed. MS analyses which exceed the control limits must be re-prepared and reanalyzed. Any problems encountered, as well as any corrective actions taken, must be reported in the case narrative.
Laboratory Control Standard (LCS)	Analyze independently prepared check sample at least every 15 samples.	For aqueous samples within the statistically determined control limits of the expected value. (Recovery limits of < ±10 %). For sediment %R- 80-120%.	The LCS must be analyzed using the same sample preparation, analytical method, and QA/QC procedures employed for the samples. If the LCS results fall outside the control limits, the problem corrected, and the samples associated with the out of control LCS reanalyzed.
Rinsate Blank	Not to exceed one Rinsate blank per day of sampling, but at least one weekly	< RL	Any problems with the Rinsate blank will be addressed by the data validator, not the laboratory.
Field Duplicates	With each batch of samples	RPD ≤ 50%; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Acid Volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM)

EPA 821-R-91-100, Draft Analytical Method for Determination of Acid Volatile Sulfide and Selected Simultaneously Extractable Metals in Sediment, December 1991

Audits Required	Frequency of Audits	Limits	Action
Required QC for AVS			
Calibration verification for colorimetric or ion-selective electrode options using known sodium sulfide QCS standard.	At the beginning and end of each batch of samples.	Per EPA 821-R-100, Section 9, analysis of known sodium sulfide QC standard should give recoveries of 85-115% of the expected.	A QCS must be analyzed immediately initially after calibrations and at the beginning and end of each batch of samples. Analysis of the QCS for AVS must have a recovery of 85-115%. If the QCS confirms that the calibration is outside limits, the problem needs to be investigated and corrected and if necessary the instrument recalibrated. Samples need to be reanalyzed.
Laboratory Reagent Blank (LRB)	With each batch of samples	<RL	An aliquot of reagent water is treated as a sample and exposed in the same manner as samples to the lab environment. Date produced is used to assess contamination form the lab environment. If values exceed the RL, laboratory or reagent contamination should be suspected and corrective action taken.
Laboratory Fortified Blank (LFB)	With each batch of 20 samples or less	%R - 80-110%	An aliquot of reagent water with a known quality of the analyte, from a source different than used for calibration, is added in the lab. The LFB is analyzed exactly like a sample to determine if the mythology is in control and the lab can produce accurate and precise measurements. If recovery is outside 80-115%, the source of the problem must be identified and resolved before continuing analysis.
Laboratory Fortified Sample Matrix (LFM)	To a minimum of 10% of samples or per set of 20 samples, which ever is greater.	%R - 80-110%	The LFM is an aliquot of an environmental sample to which a known quality of analyte is added in the laboratory. An LFM must be analyzed with a minimum of 10% of samples and is used to determine whether sample matrix contributes bias to the analytical results. If the recovery calculated per section 10.4.2 of the method is outside the recovery range of 85-105% and laboratory performance is in control, the recovery problem encountered with the LFM is than judged to be either matrix or solution related not system related. Document the problem.
Field Duplicate	With each batch of samples	RPD $\leq$ 50%, evaluated for analytes > 5 times the MDL	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.
Rinsate Blank	Not to exceed one Rinsate per day of sampling, but at least one weekly	<RL	If contamination is detected, the sampling coordinator should be notified so corrective action can be taken before the next sampling event.
Required QC for SEM			
Method Blank	per batch	<RL	The method blank must contain all the reagents in the same volumes as used in the processing of the samples. If the method blank is above the detection limit investigate the problem prior to analyzing samples.
Calibration Blank	Immediately following calibration and every 10 samples and at the end of the run.	Within three times the instrument detection limit (IDL)	Used to flush the sample between standards and samples. .Flush the system until the acceptance criteria is met.
Calibration Verification	Every 10 samples	within 15% of expected value	The results of the check sample used to verify calibration should agree within 15% of the expected value; if not terminate the analysis, correct the problem, and recalibrate the instrument and repeat analyses since the last acceptable check sample.
Matrix Spike Matrix Spike Duplicate	per batch per batch	Recovery within $\pm$ 25% of actual value RPD $\leq$ 20%; evaluated for analytes >5 times the MDL.	If the recovery limits are not met the problem should be investigated and corrected. If the acceptance criteria (% recovery and RPD) for the MSD are not met the problem must be investigated. Refer to sections 8.4 and 8.5 of method 6010C.
Interference check sample	At beginning of each analytical run	$\pm$ 20% of true value	Results should be within +20% of the true value. It they exceed this value investigate and correct the problem before proceeding with the analysis.

Total Phosphate and Orthophosphate

EPA Method 365.2, Phosphate, Orthophosphate (Colorimetric, Ascorbic Acid Method)

Audits Required	Frequency of Audits	Limits	Action
Method Blank	Per matrix, at least one per batch of 20 samples or less	< RL	Perform method blank using reagent water following the exact procedure used for field samples. If the method blank is above the detection limit investigate the problem prior to analyzing samples.
Laboratory Duplicates	Per matrix, at least one per batch of 20 samples or less	RPD ≤ 20%; evaluated for analytes >5 times the MDL.	Duplicate analyses which exceed the control limits must be reported in the case narrative.
Matrix Spike (MS)	Per matrix, at least one per batch of 20 samples or less	85-115 %R	MS analyses which exceed the control limits must be re-prepared and reanalyzed. Any problems encountered, as well as any corrective actions taken, must be reported in the case narrative.
Laboratory Control Standard (LCS)	Per batch of samples	± 10% of the expected value	An LCS should be processed with each batch of samples. If the standards does not agree within ±10% of the true value investigate the problems. It maybe necessary to prepare are a new calibration. The samples associated with the out of control standard should be reanalyzed.
Field Duplicate	With each batch of 20 samples	RPD ≤ 50%; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.
Rinsate Blank	Not to exceed one Rinsate blank per day of sampling, but at least one weekly	< RL	Any problems with the Rinsate blank will be addressed by the data validator, not the laboratory.

Nitrogen, Kjeldahl

EPA Method 351.3, Total Nitrogen, Kjeldahl (Colorimetric; Titrimetric; Potentiometric)

Audits Required	Frequency of Audits	Limits	Action
Method Blank	Per matrix, at least one per batch of 20 samples or less	< MDL	Perform method blank using reagent water following the exact procedure used for field samples. If the method blank is above the detection investigate the problem prior to analyzing samples.
Duplicate	Per matrix, at least one per batch of 20 samples or less	For water RPD ≤ 20%; evaluated for analytes >5 times the MDL. For sediments RPD ≤ 35%; evaluated for analytes >5 times the MDL.	Duplicate sample analyses which exceed the control limits must be reported in the case narrative.
Matrix Spike (MS)	Per matrix, at least one per batch of 20 samples or less	75-125 %R	MS analyses which exceed the control limits must be re-prepared and reanalyzed. Any problems encountered, as well as any corrective actions taken, must be reported in the case narrative.
Laboratory Control Standard (LCS)	At least one per batch of 20 samples or less	90-110 %R	The LCS must be analyzed using the same sample preparation, analytical method, and QA/QC procedures employed for the samples. If the LCS results fall outside the control limits, the analyses must be stopped, the problem corrected, and the samples associated with the out of control LCS reanalyzed.
Field Duplicate	With each batch of 20 samples	RPD ≤ 50%; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Ammonia

EPA Method 350.2

Audits Required	Frequency of Audits	Limits	Action
Linear Calibration Range	Initially and every 6 months	± 10% linearity	If any verification data exceeds the initial values by ±10%, linearity must be reestablished.
Method Blank (MB)	With each batch of samples	≤ MDL	An aliquot of reagent water is treated as a sample and exposed in the same manner as samples to the lab environment. Data produced is used to assess contamination form the lab environment. If values exceed the MDL, laboratory or reagent contamination should be suspected and corrective action taken.
Fortified Blank (FB)	With each batch of samples	%R – 90-100%	An aliquot of reagent water with a known quality of ammonia, from a source different than used for calibration, is added in the lab. The LFB is analyzed to determine if the method is in control and the lab can produce accurate and precise measurements. If recovery is outside 90-100%, the source of the problem must be identified and resolved before continuing analysis.
Instrument Performance Check Solution (IPC)	Immediately following the daily calibration, after every 10 samples and at the end of the sample run.	Verify that the instrument is within ±10% of calibration.	An IPC (a mid-range check standard) and a calibration blank must be analyzed immediately after daily calibration, after every 10th sample and at the end of a sample run. Analysis of the IPC must verify calibration within ±10%. If the calibration is outside limits, the IPC solution must be reanalyzed. If the second analysis of the IPC confirms that the calibration is outside limits, sample analysis must be discontinued and the cause determined. All samples following the last acceptable IPC solution must be reanalyzed.
Matrix Spike	For 10% of the samples.	%R - 90-100%	The MS is an aliquot of an environmental sample to which a known quality of analyte is added in the laboratory. An MS must be analyzed with a minimum of 10% of samples and is used to determine whether sample matrix contributes bias to the analytical results. If the recovery calculated per EPA 350.1 section 9.4 is outside the recovery range of 90-100% and laboratory performance (Section 9.3) based on analyses of a MB, a FB and a ICP is in control, the recovery problem encountered with the MS is judged to be either matrix or solution related not system related. Document the problem in the case narrative.
Quality Control Standard (QCS).	Quarterly	±10% of established QSC value.	Accuracy can be assessed by analysis of QCS. If the QCS is not within the acceptance criteira investigate the problem.
Field Duplicate Samples	With each batch of samples	RPD ≤ 50%; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Chlorophyll a

Standard Method 10200-H

Audits Required	Frequency of Audits	Limits	Action
LRB	With each batch of samples of the same matrix	≤ IDL	The lab must analyze at least one blank filter with each sample batch. The LBR should be the last filter extracted. LBR data are used to assess contamination from the laboratory environment. LBR values that exceed the IDL indicate contamination from the laboratory environment. When LRB values contribute 10% of more of the analyte level determined for a sample, fresh samples or field duplicates must be analyzed after the contamination has been corrected and acceptable LRB values have been obtained.
QCS	With each batch of samples	± 5 of the expected values	Accuracy can only be assessed by analyzing check standards as samples and QCS. Since there are no commercially available QCSs, dilution of a stock standard of a different lot number from that used for preparation of the calibration solutions may be used. Analysis of the QCS must be within ±5% of the expected value. If outside limits the problem should be investigated and corrected before results are reported.
Duplicate	With each batch of samples	RPD ≤ 20% evaluated for analytes >5 times the MDL.	Duplicate analyses which exceed the control limits must be reported in the case narrative

Chemical Oxygen Demand

USEPA Method 410.4, COD Chemical Oxygen Demand

Audits Required	Frequency of Audits	Limits	Action
Initial Calibration Check	Daily, prior to sample analysis	Within ± 10% of the expected values	Prior to analyzing samples, check calibrations by analyzing a Quality Control Standard (QCS). Sample analysis should not be reported until the control limit is met. If outside limits, re-calibrate instrument and repeat QCS.
Duplicate	1 per batch of samples	RPD ≤ 25%; evaluated for analytes >5 times the MDL.	Duplicate sample analyses which exceed the control limits must be reported in the case narrative.
On-going calibration verification using QCS	1 per batch of samples	Within ± 10% of the expected values	If the QCS control limits are not met, the analysis must be stopped and the problem corrected. The meter should be re-calibrated and all the samples since the last compliant QCS will be reanalyzed.

Biochemical Oxygen Demand

USEPA Method 4O5.1, Biochemical Oxygen Demand 5

Audits Required	Frequency of Audits	Limits	Action
Lab Duplicate	1 per batch of samples	RPD ≤ 25%; evaluated for analytes >5 times the MDL.	Duplicate analyses which exceed the control limits must be reported in the case narrative.
Field Duplicate	1 per batch of samples	RPD ≤ 50%; evaluated for analytes >5 times the MDL.	The laboratory will not know which sample is the field duplicate; if the limits are exceeding for the field replicate, this will be addressed by the data validator.

Total Dissolved Solids

USEPA Method 160.1 Method for Chemical Analysis of Water and Wastes, EPA600/4/79/020

Audits Required	Frequency of Audits	Limits	Action
Method Blank	1 per sample batch [a]	Detection limit	The method blank must follow the exact analytical procedure as the field samples. All positive sample results must be associated with an acceptable blank. If the method blank exceeds the control limit, the instrument should be recalibrated and the method blank re-prepared and re-analyzed. The blank acceptance criteria must be met prior to sample analysis.
Matrix Duplicate [b]	1 per sample batch	RPD 25 % or Diff detection limit	Duplicate sample analyses, which exceed the control limits, must be reported in the case narrative

Suspended Sediment

USEPA Method 160.2 Method for Chemical Analysis of Water and Wastes, EPA600/4/79/020

Audits Required	Frequency of Audits	Limits	Action
Method Blank	1 per sample batch [a]	Detection limit	The method blank must follow the exact analytical procedure as the field samples. All positive sample results must be associated with an acceptable blank. If the method blank exceeds the control limit, the instrument should be recalibrated and the method blank re-prepared and re-analyzed. The blank acceptance criteria must be met prior to sample analysis.
Matrix Duplicate [b]	1 per sample batch	RPD 25 % or Diff detection limit	Duplicate sample analyses, which exceed the control limits, must be reported in the case narrative

Volatile Suspended Solids

USEPA Method 160.4 Method for Chemical Analysis of Water and Wastes, EPA600/4/79/020

Audits Required	Frequency of Audits	Limits	Action
Preparation Blank	1 per sample batch [a]	Detection limit	The preparation blank must follow the exact analytical procedure as the field samples. All positive sample results must be associated with an acceptable blank. If the preparation blank exceeds the control limit, the instrument should be recalibrated and the preparation blank re-prepared and re-analyzed. The blank acceptance criteria must be met prior to sample analysis.
Laboratory Duplicate [b]	1 per sample batch	RPD 20 % or Diff detection limit	Duplicate sample analyses, which exceed the control limits, must be reported in the case narrative.

Corrosivity (pH)

USEPA SW-846-9045C

Audits Required	Frequency of Audits	Limits	Action
Instrument Calibration	Daily, prior to sample analysis	± 0.05 pH units	Each instrument/electrode must be calibrated daily with each set of samples analyzed. The initial calibration sequence must consist of a minimum of two (2) standards which bracket the expected pH of the samples and are approximately three or more pH units apart. Sample analysis cannot begin until the control limit is met. If a sample pH is at or exceeds the highest calibration standard, or is at or below the lowest calibration standard, the laboratory must recalibrate the instrument using two points which bracket the pH of the sample.
Lab Duplicate	1 per 10 samples	RPD $\leq$ 25%	Duplicate analyses which exceed the control limits must be reported in the case narrative.
Mid-range check standard	1 per 10 samples	± 0.05 pH units	If the mid-range check standard control limits are not met, the analysis must be stopped and the problem corrected. The calibration should be verified and the instrument re-calibrated if necessary. After the problem is corrected all the samples since the last compliant mid-range check standard will be reanalyzed.

Radiological Parameters

HASL-300 EML or EPA 600 4 80-032

Audits Required	Frequency of Audits	Limits	Action
Instrument Calibration	Yearly	Must be done at least annually	The detectors must be calibrated with a mixed energy standard (approximately 300 - 1800 keV) to obtain the counting efficiency vs. energy curves. A plot of the efficiency curves for all geometries should result in a smooth log-log curve. In addition, the laboratory must participate in an
Calibration Verification	Weekly	Gamma Spectroscopy: Detector Resolution – within ± 0.4 Full Width at Half Maximum (FWHM) Energy - within ± 1 keV of the known energies Efficiency - 90 – 110% of the efficiency determined during the initial calibration Alpha Spectroscopy: Detector Resolution - within ± 2% or 100 keV Energy - within " 25 keV of the initial energy determined at time of calibration Efficiency - 90 - 100% of the efficiency determined during the initial calibration	If the calibration verification does not meet the required limits, analysis must be stopped and the problem corrected. The instrument will then be recalibrated, and the calibration verified. Sample analysis cannot begin until the control limits are met.
Detector Background	Monthly/Weekly	Gamma Spectroscopy - ± 3 standard deviations of the long-term average Alpha Spectroscopy - ± 3 standard deviations of the previous background	Detector background must be performed monthly, at a minimum. The detector background criteria must be met prior to the start of sample analysis.
Chemical Tracer Recovery	1 per SDG	50 - 100 % R - Required for Alpha Spectroscopy Only	Chemical Tracer Recovery analyses which exceed the control limits must be re-prepared (as applicable) and re-analyzed one time only, with all results being reported. Any problems encountered, as well as any corrective actions taken, must be reported in the case narrative. It should be noted that the tracer solutions cannot be prepared more than two years prior to the sample analysis date.
Laboratory Control Sample	1 per sample set per matrix	Sediment Samples - 70 - 130 %R Required for Alpha Spectroscopy Only	If the LCS results fall outside the control limits, the analysis must be stopped and the problem corrected. All samples associated with the out of control LCS will be reanalyzed.
Laboratory Duplicate	1 per 20 samples per matrix	RPD≤ 35% or Difference ≤ 2 X detection limit	Duplicate analyses that exceed the control limits must be re-prepared (as applicable) and reanalyzed one time only, with all results being reported. Any problems encountered, as well as any corrective actions taken, must be reported in the case narrative.

Cation Exchange Capacity

SW-846, Method 9081, Cation-Exchange Capacity of Soils (Sodium Acetate) plus any modifications needed to prepare sediments

Audits Required	Frequency of Audits	Limits	Action
Method Blank	1 per sample batch	≤ detection limit	All positive sample results must be associated with an acceptable method blank. If the method blank exceeds the control limits the instrument should be recalibrated and the preparation blank re-prepared and reanalyzed. The blank acceptance criteria must be met prior to sample analysis.
Duplicate Analysis	1 per sample batch	RPD ≤ 20%	Duplicate analyses which exceed the control limits must be re-prepared and reanalyzed one time only, with all results being reported. Any problems encountered as well as any corrective actions taken must be reported in the case narrative.
Lab Control Standard	1 per sample batch	80 - 120 % R	The LCS must be analyzed using the same sample preparations, analytical methods, and QA/QC procedures employed for the samples. If the LCS results fall outside the control limits, the analyses must be stopped, the problem corrected, and the samples associated with the out of control LCS reanalyzed.

Geotechnical Tests

Moisture

ASTM D 2974, Standard Test Method for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils – Test Method A

Audits Required	Frequency of Audits	Limits	Action
Lab Duplicate	1 per batch of samples	RPD ≤ 20% or Diff ≤ DL	Duplicate analyses which exceed the control limits must be reported in the case Narrative.

Grain Size

ASTM D422, Standard Test Method for Particle-Size Analysis of Soils (Sieve and Hydrometer) or D 4464 Standard Test Method for Particle Size by Laser Light Scattering

Audits Required	Frequency of Audits	Limits	Action
Follow criteria included in ASTM D422 or D4464.			Follow criteria included in ASTM D422 or D4464.

Density (Specific Gravity)

ASTM D854, Standard Test Method for Specific Gravity of Soil Solids by Water Pyconmeter

Audits Required	Frequency of Audits	Limits	Action
Follow criteria included in ASTM D854.			Follow criteria included in ASTM D854.

Atterberg Limits

ASTM D4318, Standard Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils

Audits Required	Frequency of Audits	Limits	Action
Follow criteria included in ASTM D4318.			Follow criteria included in ASTM D4318.

Attachment 4

SOP No. 1: Procedure to Conduct Sample Management for CLP and non-CLP Samples

Title: Procedure to Conduct Sample Management for CLP and non-CLP Samples

I. Introduction

This guideline is to provide reference information on sample management procedures.

II. Definitions

Contract Laboratory Program (CLP). The U.S. Environmental Protection Agency (USEPA) CLP was developed to retain laboratory services that will ensure that all environmental samples collected under the Superfund Program will be analyzed in accordance with recognized EPA laboratory methods and quality assurance/quality control (QA/QC) procedures.

Target Compound List (TCL). This is a list of organic compounds typically analyzed for by the CLP. The list is broken into three subdivisions; volatiles, semi-volatiles and pesticide/PCBs.

Target Analyte List (TAL). This is a list of inorganic parameters typically analyzed for by the CLP. Parameters on this list include heavy metals and cyanide.

Routine Analytical Services (RAS). Laboratory analysis for substances or parameters shown on the TCL and TAL in solid and aqueous samples.

non-RAS. Laboratory analysis for substances or parameters not shown on the TCL and TAL. Analysis of non-soil/sediment, nonaqueous matrices, and analysis of RAS compounds using non-RAS protocols.

Trip Blanks. Trip blanks are used to check for sample contamination originating from sample transport and shipping, as well as from site conditions. Trip blanks are necessary when aqueous environmental samples are collected for volatile organic analysis and when SPMD samples are collected.

Rinsate Blanks. Rinsate blanks, also known as field blanks, are used to check the efficacy of sampling equipment decontamination procedures. Rinsates are collected for each type of sampling equipment used on site. Demonstrated analyte-free water is poured over the equipment and collected into containers and analyzed for the analytes of concern.

Environmental Duplicate. These are two separate samples collected at the same sampling point. Environmental duplicates are used to determine field sampling precision and are collected at a set frequency for each analyte group. For VOC samples, duplicate samples are

collocated samples. For all other parameters, a sample aliquot is homogenized and split into two sampling containers.

Matrix Spike/Matrix Spike Duplicates (MS/MSD). This is the process by which standard mixes of various organic TCL compounds are added to environmental samples prior to extraction. The sample is split into duplicates and analyzed. The analysis is used to evaluate the matrix effect of the sample upon the analytical methodology. Triple volume of aqueous samples for MS/MSD analysis is collected in the field, at a frequency of at least 5 percent per matrix/concentration. No extra volume is required for the soil samples.

Matrix Spike/Matrix Duplicates (MS/MD). The spike analysis is the process by which standard mixes of various inorganic TAL parameters are added to environmental samples prior to digestion. The analysis is used to evaluate the matrix effect of the sample upon the analytical methodology. The duplicate analysis in the process where the assigned sample is split in two and analyzed at the laboratory. The analysis is an indicator of a laboratories analytical precision based on each sample matrix. Double volume of aqueous samples for MS/MD analysis is collected in the field, at a frequency of at least 5 percent per matrix/concentration. No extra volume is required for soil samples.

Low-Concentration Sample. Samples in which a compound may be present at concentration levels less than 10.0 ppm.

Medium-Concentration Sample. Samples in which a compound may be present at concentration levels equal to or greater than 10.0 ppm to as much as 15 percent 150,000 ppm) of the total sample.

High-Concentration Sample. Samples in which a compound may be present at concentration levels greater than 15 percent (150,000 ppm) of the total sample.

III. Guidelines

The purpose of sample management is to assure that all samples collected during this hazardous waste site investigation are accounted for when the project is completed. The sample management officer is also responsible for assuring that the proper quality assurance/quality control (QA/QC) samples are collected. These purposes are achieved by adhering to the following procedures:

1) Laboratory Coordination

a) CLP Samples

Prior to collecting any samples, a request must be made through RSCC for a laboratory. At this time, any requested modifications to the CLP SOWs must also be described (*e.g.*, lower detection limits, adding a parameter, such as titanium, to the

TAL, requesting a quicker turnaround time (TAT)). A description of how to request CLP services is including in Section 2.4 of USEPA's CLP Guidance for Field Samplers, OSWER 9240.0-35, August 2004. A request for CLP services includes the following:

i) Contact RSCC to obtain CLP sample numbers – these are unique numbers used to identify each sample. For this project, a large block of CLP numbers will be set aside by RSCC prior to beginning sampling. Therefore, it is likely that these numbers will only need to be requested once. Refer to Attachment 1 for a memo describing some modifications to the CLP that were agreed to by RSCC for the Lower Passaic River Restoration Project.
ii) Fill out an RSCC request form. This must be sent to RSCC by 12:00 pm on the Tuesday prior to week of the sampling event.
iii) RSCC will contact the originator of the request by Friday with the Case Number and assigned laboratories. At times, the USEPA-DESA Laboratory will choose to perform all or part of the analysis requested.
iv) Since this is a long-term project, weekly contact will be maintained with RSCC.

b) Non CLP Samples

Two prime subcontractor laboratories will be procured for the Lower Passaic River Restoration project to conduct analysis of non-CLP parameters. Weekly contact must be maintained with these laboratories to inform them of upcoming sampling.

2) <u>Preparing the Sample Containers</u>

a) Malcolm Pirnie will purchase certified clean sample containers from an approved supplier. Copies of these certifications will be brought to the site while sampling and then kept in site files for future reference.

b) Each bottle used to collect a sample must be identified by a supplier and lot number to ensure that it is permanently associated with the sample collected in that particular container. This procedure also applies to containers used to carry demonstrated analyte-free water to be used for blank preparation. This is to ensure that for all samples collected, the specific sample bottles used can be traced to the sample container contractor, QC certification paperwork and custody records applicable to their identifying lot numbers.

3) <u>QA/QC Samples</u>

a) <u>VOC Trip Blanks</u>
i) One trip blank is required for each day that aqueous environmental samples are collected for volatile analysis.

ii) Trip blanks are only necessary for aqueous environmental samples. If rinsates are the only aqueous samples collected, then a trip blank is not necessary.
iii) Trip blanks consist of two 40 mL septum vials into which 4-5 drops of 1:1 hydrochloric acid (HCl) is introduced prior to filling them with demonstrated analyte-free water.
iv) Trip blanks are prepared in the field in the clean zone. They then remain with the field personnel throughout the sampling event and are shipped with the volatile cooler. Every aqueous environmental sample cooler must contain a trip blank in it.
v) The trip blank must be stored away from solvents and must be preserved, packaged, cooled to 4-6°C and shipped to the laboratory with the other aqueous samples.

b) SPMD Trip Blanks
i) One SPMD trip blank is required for each day that SPMD samples are either deployed or collected.
ii) The SPMD trip blank consists of a non-deployed SPMD that is taken to the sampling locations and opened for the same amount of the time as the SPMD sampling devices.
iii) The SPMD trip blank is analyzed for the same parameters as the SPMD environmental samples.

c) Rinsate Blanks
i) Rinsate blanks are collected for each type of equipment used to collect samples. The rinsates will be collected at a timed frequency depending on the sample capacity. At a minimum, rinsates have to be collected at one per week. At a maximum, rinsates have to be collected at one per day. Decontaminated equipment must be properly stored in an area and in a manner that will prevent cross contamination.
ii) Where possible, composite rinsates will be collected from all equipment associated to a particular matrix for analysis of non-volatile parameters. A separate rinsate will be collected for each type of equipment associated to a particular sample matrix which will be analyzed for volatile organics.
iii) Rinsate blanks consist of pouring demonstrated analyte-free water over clean equipment and collecting it into sample containers to be analyzed for the analytes of concern.
iv) Rinsate blanks are preserved, packaged, and shipped in the same manner as low concentration aqueous environmental samples.

d) Environmental Duplicates
i) Samples for duplicate analysis are collected in the field, for each matrix sampled at a frequency as described in Lab Task Order.
ii) Sufficient quantity of matrix must be collected from the same sample location to fill a duplicate set of sample containers. The duplicate volume is shipped to the laboratory under a separate CLP sample number.
iii) For soil/sediment samples the volatile organic fraction is collected as collocated grab samples while the non-volatile fraction is homogenized prior to collection.

e) Matrix Spike/Matrix Spike Duplicate (MS/MSD) & Matrix Spike/Matrix Duplicate (MS/MD)

i) The designation of a sample for MS/MSD analysis for organics and MS/MD analysis for inorganics is required for 1 in 20 environmental samples per concentration/matrix.

ii) Three times the total volume is necessary for collection of aqueous MS/MSD organic samples. Two times the total volume is necessary for collection of aqueous inorganic MS/MD samples. No extra volume is required for the soil samples.

iii) MS/MSD and MS/MD samples are noted as such on the chain of custody (COC).

4) Sample Documentation, Packaging, and Shipping Procedures

One or more of the field personnel will be designated as the sample management officer(s). The sample management officer will bear the ultimate responsibility for the documentation, packaging, and shipping of the samples. These procedures are outlined below.

a) Documentation/Chain of Custody

For documentation purposes, the field team will enter information about each sample into the field laptop as they collect the sample. As this information is entered into the laptop, it is transmitted to the PREmis database. Information recorded includes the following:

- Sample date and time of collection
- Associated QC samples
- Analyses required
- Bar code number – since the bottles do not receive sample labels until they are returned to the field office, a sample bar code is placed on each bottle when the samples are collected. This information is entered into the field application so the bar code is permanently associated with a specific sample bottle.

i) Since all of the sampling information is recorded electronically the sample management officer can electronically generate the COC and sample labels. The sample management officer needs to access the sample management PREmis module. This will allows the sample management officer to designate which samples are in which shipment. This is required since there will be numerous laboratories for this project.

ii) Once all of the samples are associated to a shipment, the COC and sample labels can be printed from PREmis. The sample labels are affixed to each sample container and covered with clear tape. In addition, for CLP samples, a sample label is placed on the sample tag. The sample labels will contain the following information:

- MPI-designated sample number
- For CLP samples only, the assigned CLP Number
- The month, day, and year the sample was collected
- The type of analysis requested
- The type of preservation performed in the field.

b) Packaging and Shipping Samples

i) Make sure the caps on the sample bottles are tightly sealed. Wipe down the outside of all of the sample bottles.

ii) Preserve the samples according to the SOP No. 2 for Sample Preservation.

iii) Apply one custody seal around the circumference of the container or over the cap and onto the sides of the container. The custody seal must applied to sample containers in such a manner as to reveal if the container was opened during transit. Note: Septum vials should not be covered over the top.

iv) Place each container in its own ziplock bag. The two 40 ml vials may be placed in one bag. Eliminate extra air space from the bag before sealing. The EnCore® device comes in its own ziplock bag and this bag will be used.

v) For CLP samples, place the associated sample tag into the ziplock bag with the sample.

vi) Prepare the shipping container (usually a cooler). The cooler will be prepared so that no leakage can occur during shipping. All valves on the cooler will be securely duct taped, both inside and outside the cooler, and the cooler will be lined with either plastic or a large garbage bag. Only coolers that conform to the general design requirements in 49 CFR 173.410 will be used for shipment.

vii) The VOC samples should be packed together, without any other sample fraction, with the trip blank.

viii) Put 1-2 inches of packing material in the bottom of the coolers, then place the samples into the cooler.

ix) Surround the sample bottles with bags of ice (only the samples that need to be cooled – Refer to the SOP for Sample Preservation No. 2. The ice will not be kept in its original bag, but will be repacked into ziplock bags. Use enough ice to ensure that the proper temperature (4-6°C) is maintained during transport. Place a temperature blank (40-mL vial filled with DI water) into the cooler.

x) Place packing material over and around the sample bottles. Sufficient packing material must be used to the bottles will not move or break during transport.

xi) Once the samples are packed, the plastic or garbage bag will be closed and securely taped.

xii) Prior to shipment the relinquished by and received by sections of the COC form will be filled in. Generally, the shipper will not sign the COC. Therefore, the carrier's name is filled in by the sample management officer. The original COC form will then be placed in a ziplock bag and taped to the inside of one of the lead cooler; one copy of the COC form(s) will be placed in a ziplock bag(s) and placed in the other cooler(s).

xiii) For CLP samples, one copy of the COC form will be retained by the sample management officer and one copy will be sent to RSCC. For non-CLP samples, one copy of the COC form will be retained by the sample management officer.

xiv) Close the cooler and seal with strapping tape. If visibly dirty, the outside of the cooler will be wiped down. Apply signed and dated custody seals to the cooler. Place two custody seals diagonally across from each other where the cooler lid meets the cooler. The custody seals will be applied in such a manner as to reveal if the cooler was opened during transit.

xv) An address label will be placed on the outside of each cooler. The label will be covered with clear tape. If more than one cooler is being sent to one destination, each cooler will be appropriately labeled as 1 of X, 2 of X, *etc.* The airbill will be attached to one of the coolers. Usually, the samples will be sent via overnight carrier for next day delivery. This should be confirmed with the Field Team Leader.

xvi) The laboratory will be notified of the shipment before 9 a.m. ET on the day after shipping. For CLP samples, fill out the Sample Shipping Call-In Form. Call or fax the shipping information to RSCC by 9:00 am the following morning. For non-CLP samples, the notification system agreed to in the subcontract will be followed.

Note: Some samples have very short holding times. In some limited instances, the samples may be either hand delivered to a laboratory or picked up by the laboratory's courier service.

ATTACHMENT 1

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION II

DATE: January 14, 2004

SUBJECT: Request for Modifications of CLP Requirements for the Lower Passaic River Restoration Project

FROM: Jennifer E. Feranda, CLP Project Officer/RSCC Coordinator
Hazardous Waste Support Section (2DESA-HWSB)

TO: Alice Yeh, Remedial Project Manager
2ERRD

The purpose of this memorandum is to follow up on your letter of July 25, 2003 and sub-sequent phone conversations concerning the request for modifications of Contract Laboratory Program (CLP) requirements for the Lower Passaic River Restoration Project. Below, I have outlined your specific requests as well as provided HWSB response(s) as to whether or not these requests can be accommodate.

If you have any questions or would like to discuss this in more detail, please do not hesitate to call me at (732) 321-6687.

Response to Requests for Modifications of CLP Requirements for the Lower Passaic River Restoration Project

Request for Modification to FORMS II Lite Application Requirement

1) ***Request:*** Malcolm Pirnie (MPI) has developed a web-based data management system named PREmis (the Passaic River Estuary management information system) to handle existing historical data and new data collected for the Remedial Investigation/Feasibility Study (RI/FS) of the Lower Passaic River. PREmis contains all the fields required by FORMS II Lite, but also has numerous additional data requirements associated with the unusually complex modeling effort planned for the Lower Passaic River Restoration Project. It was requested that the use of PREmis be granted in lieu of the use of FORMS II Lite. Information contained in the PRE mis database would be directly copied into the FORMS II Lite database, thereby satisfying the FORMS II Lite reporting requirements.

Response: PREmis can be used for the project, however, it can not be used in lieu of FORMS II Lite. Traffic Reports/Chain of Custody (TR/COC) forms that accompany samples to the laboratories will need to be generated by FORMS II Lite. In addition, either the XML files with information from the FORMS II Lite database or hard copies of the TR/COCs will need to be transmitted to the CLP's Sample Management Office (SMO) on a pre-determined schedule (within a day or two of sample shipment).

Request for Modifications to the Contract Laboratory (CLP) Requirements

2) ***Request:*** A specific cohort of laboratories (both organic and inorganic) would be assigned to the project for the duration of the Remedial Investigation sampling program (several years) prior to the beginning of sampling. The Passaic River Estuary project team would determine which laboratories receive specific samples.

Response: This request can not be accommodate. Due to laboratory capacity, laboratory performance, and turn over of contracts, specific labs can not be committed to an entire project. The frequency that laboratory space is booked and the length of time that a lab or labs can be utilized will be determined as we get closer to the actual sampling event. Based on the number of labs being used and their capabilities per their contracts, the Lower Passaic River project team may or may not be able to determine what labs receive specific samples (*e.g.*, if there are two labs assigned, one organic and one inorganic, organic samples must go to the organic lab)

3) ***Request:*** All sample log-in information would be entered into the PREmis Website by the laboratory instead of onto hard copy log-in sheets.

Response: Due to the requirements and constraints of the CLP contracts, this request will not be able to be accommodated at this time.

4) ***Request***: A large block of sequential CLP number, both organic and inorganic, would be designated specifically for this project.

Response: Starting and ending CLP sample numbers will be assigned for this specific project. PREmis can be used to generate a large block of sequential CLP sample numbers, both organic and inorganic as needed during the project.

5) ***Request:*** Laboratories would be required to submit EDDs according to project specific standards in a timely manner, usually with the hard copy of the CLP package. If the EDD format were incorrect, the laboratory would need to submit a corrected EDD.

Response: Electronic data deliverables (EDDs) will be submitted to the data user(s) in the Multimedia Electronic Data Deliverable (MEDD) format. The EDDs will transmitted to the data users by EPA Hazardous Waste Support Section (HWSS) staff once data has been reviewed for contract compliance. Any incorrect or incomplete EDDs will be corrected prior to the data users receiving the files. The time frame for receipt of these deliverables will be pre-determined prior to the start of sampling for this project.

Attachment 5

SOP No. 2: Procedure to Conduct Sample Preservation

Title: Procedure to Conduct Sample Preservation

I. Introduction

This guideline is to provide reference information on the accepted methods of sample preservation.

II. Materials

Preservatives:

a. 1:1 HCl - (Hydrochloric Acid/Deionized Water)
b. HNO_3 - full strength (Nitric Acid)
c. NaOH - 10 N (Sodium Hydroxide)
d. H_2SO_4 - full strength (Sulfuric Acid)

Additional Materials:

a. Disposable Pasteur pipettes
b. Pipette pumps - 10 ml or 2 ml
c. Latex pipette bulbs
d. Squeeze bottle with deionized water
e. Clear wide mouth glass jar for water pipette
f. Paper towels
g. Lead acetate paper
h. Cadmium nitrate or cadmium carbonate (if using lead acetate paper)
i. Potassium iodide - starch test paper (KI-starch paper)
j. Ascorbic Acid (if using KI starch paper)
k. Filter paper
l. Filter funnels (disposable or decontaminated)
m. Filter vessel with hand pump
n. pH paper
o. Scale

Safety Materials:

a. 2 pair safety glasses
b. 2 pair solvex gloves
c. 2 labcoats
d. MSDS sheets
e. Eyewash

III. Discussion

Complete and unequivocal preservation of samples is a practical impossibility. At best, preservation techniques slow down the chemical and biological changes that inevitably continue after the sample is removed from the parent source. The changes that take place in a sample are either chemical or biological. In the former case, certain changes occur in the chemical structure of the constituents that are a function of physical conditions. Metal cations may precipitate as hydroxides or form complexes

with other constituents; cations or anions may change valence states under certain reducing or oxidizing conditions; other constituents may dissolve or volatilize with the passage of time; and metal cations may also adsorb onto surfaces (glass, plastic, quartz, *etc.*). Biological changes taking place in a sample may change the valence of an element or a radical to a different valence. Soluble constituents may be converted to organically bound materials in cell structures, or cell lysis may result in release of cellular material into solution. The well known nitrogen and phosphorus cycles are examples of biological influence on sample composition. Therefore, as a general rule, it is best to analyze the samples as soon as possible after collection. This is especially true when the analyte concentration is expected to be in the low ug/l range.

Methods of preservation are relatively limited and are intended generally to (1) retard biological action, (2) retard hydrolysis of chemical compounds and complexes, (3) reduce volatility of constituents, and (4) reduce absorption effects. Preservation methods not outlined below are generally limited to pH control, chemical addition, refrigeration, and freezing.

IV. Guidelines

All Samples

With few exceptions, most samples need to be cooled to between 4-6 °C immediately after sample collection.

Preserving Aqueous Volatile Organic Compound (VOC) Samples

Equipment

Field personnel should take the following materials for VOC sample preservation to the sampling locations:

1. One 40-mL VOA vial containing 1:1 HCl.

   The 1:1 HCl should be transferred on site from a 1-liter plastic-coated glass bottle to one properly labeled 40-mL glass vial by using a glass funnel. This should be performed at the field office. Hand and eye protection must be worn during the transfer and handling of hydrochloric acid. Field personnel must attempt to keep the 40 ml vial in an upright position during field sampling. The 1-liter plastic-coated bottle must be kept at the field office; the 40-mL vial must be kept in a plastic ziplock bag.
2. Plastic ziplock bag containing pH indicator strips for each sampling location.
3. Latex gloves
4. Eye protection
5. Plastic ziplock bag for disposal of used pH indicator strips and latex gloves.

Preservation Procedures

1. For each different type of aqueous sample to be collected (*e.g.*, river sample, CSO sample) a test sample must be preserved to determine if the preservation procedure will cause an adverse reaction. Note that a test vial must also be collected when the temperature changes (*e.g.*, each season) and whenever a sample is significantly different in appearance than the test sample.

First, fill a test vial one-half full with the sample matrix to be collected. Note the color and clarity of the sample.

2. Test the pH by inserting one pH paper strip into the test vial. If the pH is less than 2.0, as indicated by a blue color on the strip, collect the samples without acidifying. Document this in the field application. The field sample management officer must document the sample as not preserved on the COC. If the pH is greater than 2.0, continue to Step 3. The pH indicator paper strip should be put into a plastic bag for later disposal.
3. Dispense 10 drops of 1:1 HCl from the pipet. Tap the vial gently to mix. If color develops, precipitates form, effervescing occurs, or an exothermic reaction (heat generation determined by holding the vial firmly) occurs, do not acidify the samples and document the reason for not acidifying in the field application. This information should also be included on the COC. If none adverse reactions occur when acid is added to the sample, proceed to Step 4.
4. Test the pH of the sample. If the pH is less than 2.0, proceed to Step 5. If the pH is greater than 2.0, add 1:1 HCl a few drops at a time (keeping count) until the pH is less than 2.0; then proceed to Step 5.
5. Fill the test vial with sample until the vial is nearly full to the top. Gently tap the side of the vial to mix, and test the pH of the sample. If the pH is less than 2.0 proceed to the next step. If the pH is greater than 2.0, again add 1:1 HCl a few drops at a time (keeping count) until the pH falls below 2.0. Proceed to the next step.
6. Note the amount of 1:1 HCl added to the test vial. Add this amount of 1:1 HCl to all of the samples, using the same glass pipet, after collecting the samples, and before capping the 40 ml vials. To avoid cross contamination, the sampler must be extremely cautious not to touch the glass pipet to the sides of the vial or the sample. Document the approximate quantity of 1:1 HCl added to each sample. These samples are then packaged and cooled to 4^0C prior to shipping to the CLP laboratory.
7. Store the samples at 4^0C until the time of analysis.
8. Properly dispose of the test vials and all used sample preservation equipment.

Preserving Aqueous Inorganic Samples with Acid

1. Add the acid to the sample using a pipette. Typically, depending on the size of the pipette and the original pH of the sample, approximately ½ a pipette of acid is required per liter of sample. Recap the sample bottle and turn it gently upside down to mix the contents.
2. Check the pH by pouring an aliquot of the sample over the pH paper; do no dip the pH paper directly into the sample. The pH of the sample should be < 2.
3. If the sample contains a significant particulate fraction, acidification without filtration could result in deceptively high values for the aqueous sample. Varying amounts of particulate matter can also give large differences in metal values for duplicate acidified aqueous samples. Observation, therefore, should be made and recorded in the field application and also noted on the COC. If an obvious change is observed during sample preservation, which may bias the results, the Site Quality Control Officer (SQO) should be consulted.
3. If the pH is still > 2, repeat steps 1 and 2 until the pH is < 2.
4. Store the samples at 4^0C until the time of analysis.

Preserving Aqueous Cyanide Samples

1. Test a drop of sample with potassium iodide-starch test paper (KI-starch paper). A resulting blue color indicates the presence of oxidizing agents and the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.
2. Add NaOH to the sample using a pipette. Typically, depending on the original pH of the sample, approximately 2 mL of NaOH per liter of sample is required. Recap the sample bottle and turn it gently upside down to mix the contents.
3. Check the pH by pouring an aliquot of the sample over the pH paper; do not dip the pH paper directly into the sample. The pH of the sample should be > 12.
4. If the pH is still < 12, repeat steps 2 and 3 until the pH is > 12.
5. Store the samples at 4^0C until the time of analysis.

Refer to the sample preservation tables (3-1 to 3-6) in the QAPP for specific sample preservation requirements.

Attachment 6

Sample Receipt Checklist

Example Sample Receipt Checklist
Lower Passaic River Restoration Project

LIMS#:_____

Project:_____ Date Received:_____

Number of Coolers:_____

USE OTHER SIDE OF THIS FORM TO NOTE DETAILS CONCERNING CHECK-IN PROBLEMS

A. Preliminary Examination Phase: Date cooler was opened:_____

Person Opening Cooler: *Printed Name:*_____ *Signature:*_____

1. Did the cooler come with an airbill?..... YES NO

   If yes, enter the Carrier Name and airbill number:_____

2. Were custody seals located on the outside of the cooler?..... YES NO

   If yes, how many and where were they located?_____

   If yes, were they signed and dated?_____ Date on the custody seals?_____

3. Were custody seals unbroken and intact upon arrival to the laboratory?..... YES NO
4. Was the COC sealed in a ziplock bag and taped to the inside of the cooler?..... YES NO
5. Was the COC filled out properly?..... YES NO
6. Did the laboratory representative sign the COC in the appropriate place?..... YES NO
7. If required, were the samples cooled to the proper temperature with ice?..... YES NO

   If yes, what was the cooler(s) temperature(s) upon receipt?_____

B. Log-in Phase: Date cooler was logged-in:_____

Person Logging-in Cooler: *Printed Name:*_____ *Signature:*_____

8. What type of packing material was in the cooler?_____
9. Were all the bottles (except VOCs) sealed in separate ziplock bags?..... YES NO
10. Did all the bottles arrive unbroken and were the labels legible?..... YES NO
11. Did the bottle labels agree with the COC?..... YES NO
12. Were the correct sample containers used for the analyses requested?..... YES NO
13. Were the correct preservatives added to the samples?..... YES NO
14. Was a sufficient amount of sample sent for the analyses requested?..... YES NO
15. Were any problems with the samples discovered?..... YES NO

    If yes, was the site manager called?..... YES NO

    If yes, prepare a telephone log and attach to this form.

Attachment 7

Cape Technologies Technical Note: TN-004 Immunoassay Dioxin

CAPE Technologies

DRAFT

High Performance Dioxin/Furan Immunoassay Kit

Technical Note TN-004

Quantitation, Calibration, and Quality Assurance for Method 4025m

Quantitation: Dioxin/furan analysis by US EPA Method 4025m using the CAPE Technologies DF1 Immunoassay Kit gives quantitative results which correlate with TEQ (per Application Note AN-008). However, just as with conventional chemical analysis, proper calibration and quality assurance are required for maximum reliability.

The DF1 immunoassay is inherently quantitative. Each immunoassay run should include 2378-TCDD standards to define a standard curve as described in Section D (Table 1) of the kit insert IN-DF1. This curve is applied to unknowns using Calculation Module C, a special purpose Microsoft Excel file available from the CAPE Technologies web site (www.cape-tech.com). Module C uses an iterative non-linear curve fitting procedure based on the same four parameter equation which is the basis for a variety of commercial immunoassay data analysis software. Module C calculates the best fit standard curve and the concentrations of unknowns based on that curve. Background information and instructions are included with Calculation Module C.

The process described above produces raw quantitative results based on the standard curve, which may or may not be an acceptable endpoint. If the analyst's goal is relative quantitation (i.e. looking for hot spots- finding deviations from a certain baseline and estimating their concentration relative to that baseline), then no calibration adjustment is required. However, if the goal is absolute quantitation (as for virtually all dioxin analysis by GC-MS), then a calibration adjustment must be applied to the raw quantitative results. Calculation Module C has this calibration adjustment calculation built in, but the analyst must determine the actual calibration adjustment factor (CAF) and provide the QA data supporting its use.

Calibration of other 4000 series methods: In order to articulate the rationale supporting this calibration adjustment, it is helpful to first describe the approach to calibration for the other 4000 series immunoassay methods approved by the US EPA (www.epa.gov/epaoswer/hazwaste/test/4_series.htm). These methods, such as Method 4020 for PCBs, have a calibration adjustment built into the method. This adjustment is determined by the kit manufacturer and is applied on the front end, through the use of immunoassay calibrators instead of standards. These calibrators are designed to let the analyst make semi-quantitative decisions at pre-selected levels, such as 1, 5, 10, or 50 µg/g. Once the kit user compares the sample to a calibrator in the same run and makes a decision, no further data interpretation is required. The calibration rationale assumes that the samples to be analyzed and the decision levels to be used are the same as those used for the validation study.

The actual concentrations of these calibrators may differ from the decision level by a factor of two or more. For example, users of one of the Method 4020 PCB kits would make a decision on whether the sample PCB level is less than 10 µg/g by comparing it to a calibrator in the same run that actually contains 5 µg/g PCB. This difference between decision level and actual concentration used for the calibrator is determined by splitting samples and analyzing by both the conventional method and the immunoassay, in quantitative mode and with no adjustment of the data. The resulting quantitative relationship between the two data sets is used to set the calibrator level so that a minimum false negative rate is achieved in the semi-quantitative decision making process.

There are several good reasons why these quantitative results from the two methods might not follow a 1:1 relationship (regression line slope of 1), even if the correlation is excellent. These include, but are not limited to, reduced efficiency of the rapid extraction method, effects of differences in congener profile between the PCB in the sample and standard, and random variation. The front end calibration procedure described above allows compensation for all such factors together, without explicitly determining their individual contributions. The calibration adjustment described above is effectively the same as obtaining unadjusted quantivative results, then multiplying them by a uniform adjustment factor. The approach to calibration for Method 4025m is similar and accomplishes the same goal, but with some very important differences. The rationale for this approach is described below.

Calibration rationale and procedure for Method 4025m: The same factors noted above which can cause the regression line slope to be less than 1 must also be dealt with when calibrating Method 4025m. However, there are more potential factors because of the increased complexity of the procedure (e.g. recovery through cleanup and solvent exchange as well as extraction) and because of the greater variability of the analyte composition (congener profile) among the population of possible samples. For these and other reasons, the front end calibration approach described above for other 4000 series immunoassays is not viable for Method 4025m. Therefore Method 4025m analysis uses standards rather than calibrators, and the analyst applies a back end calibration adjustment to the raw quantitative results.

The calibration procedure supported by the above rationale is straightforward. A set of split samples is analyzed by the reference method (GC-MS) and also by Method 4025m. The comparison data set will likely have some deviation from the ideal 1:1 relationship noted above (regression line slope other than 1). A new data set of adjusted 4025m results is created by multiplying each raw 4025m result by the CAF (starting at 1). The CAF is then changed and the regression line slope is calculated for the adjusted 4025m data. The final CAF value is that which gives a regression line slope of 1 for the adjusted 4025m data. This CAF is then uniformly applied to all raw 4025m results. Once a CAF is determined, it should be checked and refined continuously using the stream of GC-MS data from ongoing quality assurance samples. On a larger project, from 5 to 20 percent of samples screened by Method 4025m should be split for conventional analysis. These are the most important quality assurance samples, but are by no means the only ones that should be run.

Notes on calibration quality: For best results, calibration adjustment should be done on a site specific basis if possible. Differences in dioxin source, sample matrix, and congener profile will all increase the variability of quantitative results and decrease the probability of success. The effect of congener profile on calibration can be estimated in advance using Calculation Module A. More samples will obviously give better results. It is theoretically possible to base a CAF on a single sample, but statistically unwise. Likewise, it is statistically best for the samples on which the CAF is based to cover as wide a concentration range as possible.

The closer the calibration samples are to the target sample population, the better the calibration adjustment will be. It is possible to use other reference samples for calibration, but the results will not be as good as when using samples from the same set as the unknowns. For example, calibration based solely on spiked samples can be used, but is less than ideal, since it will not account for extraction differences between spikes and incurred residues. Likewise, calibration based solely on unrelated samples, such as standard reference materials, will not account for matrix differences between the reference sample and the unknown samples.

Attachment 8

Cape Technologies Technical Note: TN-005 PCB Immunoassay

CAPE Technologies

DRAFT

High Performance PCB-TEQ Immunoassay Kit (PCB1)
High Performance Dioxin/Furan Immunoassay Kit (DF1)

Technical Note TN-005

Preparation of Samples for PCB-TEQ Analysis Using Carbon Column Fractionation

Existing Carbon Column Method: The preparation of samples for dioxin/furan analysis by US EPA Method 4025m is described in CAPE Technologies Application Note AN-008. This method uses a two stage coupled column system for cleanup of an extract in an aliphatic solvent (such as hexane or hexane/tetradecane). The second stage of this cleanup is an activated carbon mini-column which is used to capture the dioxin/furan portion of the sample for analysis with the DF1 Immunoassay Kit. The protocol described in Application Note AN-008 calls for loading the sample onto the carbon mini-column, washing with 6 mL of 1:1 hexane:toluene in the forward direction, then reversing the column to elute the dioxin/furan sample with 12 mL of toluene. It is very simple to modify this protocol to allow capture of the dioxin-like PCB fraction from the same sample.

Fractionation Protocol: The protocol modification noted above is as follows:

1) after removing the carbon column from its acid silica column during the sample loading (step F7/8), the column is placed on a clean empty reservoir for washing of the carbon column alone (as in the first portion of AN-008 step F9)
2) the column is washed in the forward direction with 5 mL hexane (new step)
3) the dioxin-like PCB fraction is eluted in the forward direction with 6 mL of 1:1 hexane:toluene and captured for analysis (exactly as in AN-008 step F9, except that the eluate is captured here)
4) if analysis of the dioxin/furan fraction is required, continue as normal in AN-008 (step 10); reverse elute with 12 mL toluene to obtain the dioxin/furan fraction

Analysis of Eluted PCB's: The captured dioxin-like PCB fraction is exchanged for immunoassay analysis using the same protocol as described for dioxin/furan analysis. An aliqout of immunoassay keeper is added and the sample is evaporated under a nitrogen stream with gentle heating. The residue is centrifuged and methanol is added to dilute the sample prior to addition to the immunoassay tube. The complete PCB immunoassay analysis procedure is described in detail in the PCB-TEQ Kit Insert (IN-PCB1).

Supporting Data: The original design of the carbon column method in AN-008 was intended to remove as many potentially interfering compounds as possible from the dioxin/furan sample. The protocol as outlined in AN-008 captures in the dioxin/furan fraction all the tetra- and higher chlorinated PCDD's and PCDF's which contribute to the TEQ and are detected by the DF1 immunoassay. The preceding hexane:toluene fraction described above contains the major crossreacting PCDD/F, 237-triCDD, as well as the 12 WHO dioxin-like PCBs. Other PCBs are flushed through the carbon column during the hexane washes before and after the carbon column is removed from the acid silica column, before the hexane:toluene fraction. This carbon column elution behavior has been verified using stable isotope labeled dioxin/furan and PCB congeners, analyzed by HRMS.

The CAPE Technologies PCB-TEQ Immunoassay and the fractionation protocol described above were evaluated in a 2004 demonstration project as part of the Superfund Innovative Technology Evaluation (SITE) Program. The final report is not public yet, but will be released in 2005. The EPA concluded that the PCB-TEQ kit, with the cleanup method described above, could be an effective screeing procedure for PCB TEQ. These data will be released to CAPE Technologies customers concurrent with the release of the final SITE Program Demonstration report.

Parallel Analysis of TEQ from PCDD/Fs and PCB's: The carbon column fractionation described here allows a single sample to be extracted and prepared for immunoassay analysis using both the DF1 Dioxin/Furan Kit and the PCB1 PCB-TEQ Kit. The resulting data can be combined to give a total TEQ value from PCDD/F's and PCB's, as well as defining the relative contributions of the two components. The amount of time required for this combined analysis is only marginally greater than for either analysis alone. In addition to the "piggybacked" sample preparation by carbon column fractionation, the immunoassays can be run concurrently, with slightly staggered incubation times. The potential economic and scientific benefit of this approach for assessment of either unknown sites or known PCB/dioxin sites is huge.

Attachment 9

Memorandum on Security of Field Applications

Memo

Date: 07/24/2003

To: Alice Yeh, Bruce Fidler, Rob Danowski, Ertan Akbas

From: Lisa Szegedi-Greco

RE: Security on the Field Application - Revised

The Passaic River Estuary Superfund Site consists of approximately 17 miles of the Passaic River from its mouth at Newark Bay upstream to the Dundee Dam. The study area for the site also includes the Hackensack River from its mouth upstream to the Oradell Dam, Berry's Creek, Pierson Creek, portions of Newark Bay, the Kill van Kill and the Arthur Kill. Currently, it is anticipated that sampling will begin in the study area within the next year. Due to the complexities of the site (i.e., the number of potentially responsible parties [PRPs] and trustees that are involved, the magnitude of the sampling event [i.e., thousands of surface water, sediment, and biota samples, being analyzed by numerous laboratories for a large suite of parameters], the speed at which the work will take place) it is imperative that an appropriate system should be implemented to assure that the field data collected are accurate, complete, and legally defensible. The magnitude and complexity of the sampling program would render impractical the use of traditional field data collection methods (i.e., handwritten field logbooks and data sheets). A more efficient solution that would increase the quality of the data, greatly reduce transcription errors, and allow multiple team members at various locations access to the data, is to collect and control the field data electronically. The purpose of this memo is to summarize the innovative electronic field data collection and control methods already being used by Malcolm Pirnie on behalf of USEPA and the Kansas City District at another Superfund Site to facilitate determination as to whether the system is sufficiently secure for the purposes of the Passaic River project.

Data collection occurs on a Visual Basic application (developed in-house) (with an MS Access database) that is downloaded onto a field laptop computer. The following section summarizes data collection from the field to the project website:

1. First, a secure project website is established. Security on the website consists of secure socket layers (i.e., https site), password protection, and multiple user levels. These user levels restrict access and rights to certain portions of the website. For the Passaic River project, this electronic access security would be supplemented by the existing confidentiality / non-disclosure agreements which would discourage system users from distributing their usernames or passwords to others outside the approved team. The system could also be set up to require periodic password changes.

2. Next, information needed for the field is entered onto selected pages of the website. For example, all of the field instruments (e.g., Horiba, photoionization detector (PID)) are assigned a unique barcode identifier. Information for the equipment (e.g., model, calibration date) is then entered into the project website on the equipment page.

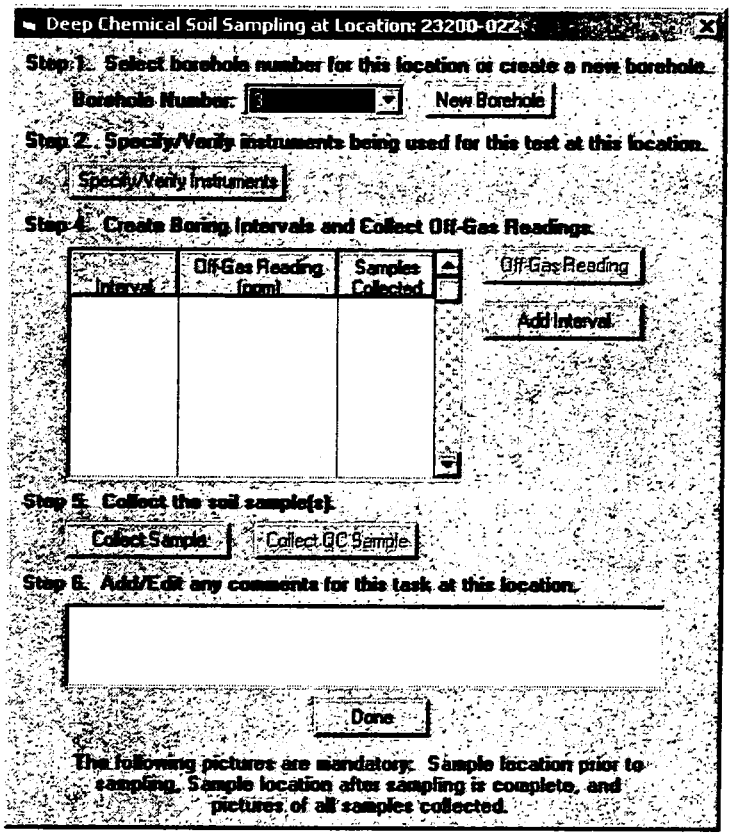
3. A calendar of field events (with a comments section) is created to assist the field team(s) with their work, and to ensure that all teams know and understand their sampling assignments. Work orders that specify where sampling is to occur, what parameters should be analyzed for, as well as any other pertinent information, are also created in the calendar.

4. When the field team(s) begins work, each team is assigned a field laptop that has a specific identification number associated with it. When the field team launches the field application the user is prompted for

their unique username and password. This way, the field application keeps a log of who entered in what information, along with the dates and times the information was entered. The purpose of this is twofold; this acts as each field team member's electronic signature and it also ensures that unauthorized users cannot access the software (i.e., write in someone else's logbook).

5. At the beginning of each new sampling event, the field team downloads a work order, that is specific to that field team, from the project website to the field laptop. The work order contains that crew's field assignment (e.g., chemical sediment sampling in the Passaic River between river miles 2.0 and 3.0), as well as information about previous sampling that occurred at this location. Each week, the field team also updates the background information associated with each work order (e.g., equipment IDs) by downloading this information from the website.

6. When the field team begins collecting sampling information, they are required to fill in a series of information windows (see example below) that consist of pick lists, comments fields, and automatically generated fields. For example, if a field team is collecting a chemical sediment sample, the field application, *not the field team*, assigns the sample ID. Since the sample ID also contains the unique identifier for the laptop from which it was requested, sample IDs are never duplicated. Another advantage is the elimination of missing information since certain fields must be filled in prior to moving to another window.



7. After the field team completes an information window and clicks the button labeled "Done," the information entered into the window can be viewed but it cannot be changed. This is analogous to the field team not being allowed to erase information once it's entered into the field logbook.

8. All the information collected in this application is written to a secure password-protected MS Access database accessible directly only by a database administrator. Since the database is secure, the field team is not able to make any changes to the records contained in it.

9. After all sample collection is complete, the field team returns to the field office to upload the information to the project website. The field team then prints out the field data collection report from the website, reviews the report, and initials and dates each page. Copies of this report are kept at the site field office under the field team leader's control

10. Once the information is on the website it is reviewed by the Site Quality Control Officer (QCO) or his/her designee. They can either accept or reject each piece of data. During this review and/or the field team's review of the report, it is possible that mistakes or omissions in the information will be noted. When this occurs, the field team is supplied with a paper form to fill out that requests either supplemental information or corrections to the data. This information is then added to the report by one of the site administrators. A complete paper record of the change and/or addition, the person requesting the correction, the person supplying the information, and the date of the change, is maintained in the site files.

Advantages of this system over traditional data collection and control methods include the following:

1. Field data are typically available for review within hours after being collected. Once the data are uploaded to the web site, any member of the project team can view the data in a standardized report format that lists the geographic location for each sample or measurement, any associated quality control, all instrument measurements and response checks, and what type of laboratory sample was collected.

2. Collecting data with this system greatly improves the quality of the data since it nearly eliminates data omissions, reduces the amount of transcription errors, and automates some field quality control (QC). The field application prompts the field team to collect QC samples (duplicates, matrix spikes (MS), matrix spike duplicates (MSD), matrix duplicates (MD), and rinsates) and it also does not allow certain incorrect information to even be entered. When using a traditional logbook, there are no checks on the information that is entered, which can result in missing or incomplete data. Given this, the data evaluation team might not discover that information was missing until several weeks after the field work was completed. At that point, recapturing the information could be costly, if not impossible. In the application it is nearly impossible to omit essential information since certain fields are mandatory and the data collection team cannot proceed through the application without completing them.

3. Instrument QC is entered directly into the system at the beginning and end of each day. If the response check indicates that the instrument is not working properly (e.g., the PID response is greater than 2 parts per million different from the standard gas concentration), the user is prompted to use a different instrument. This allows the field team to immediately identify if a problem is occurring, thus eliminating wasted field effort.

4. Quality control calculations are also built into the system. For example, when the field team collects a duplicate measurement with an instrument, the field application will calculate the relative percent difference and determine if it falls within the required limits. If not, a message will appear on the screen warning the user to check the instrument. This function virtually eliminates wasted field effort due to malfunctioning instruments.

As described above, once the field data are collected, the information is uploaded from the field application to the project website. A module on the website allows the field team to select individual samples, create chain of custody forms, and mark the samples as shipped to the laboratory. Each chain of custody form is retained electronically on the system; a signed hard copy of the form is also retained in the site files, under control of the field team leader. Once the laboratory receives the samples, a module on the website allows them to mark each shipment as received. Any problems with the shipment such as broken custody seals or insufficient sample volume, are also marked on the website.

Attachment 10

SOP No. 3 Technical System Audit and Example TSA Audit Form

Title: Procedure to Conduct a Technical System Audit (TSA)

I. Introduction

This guideline is to provide information on TSAs to be conducted for the Lower Passaic River Restoration Project.

II. Guidelines

The purpose of the TSA is to ensure that the sampling team adheres to the guidelines contained in the Work Plan, Field Sampling Plan (FSP), and Quality Assurance Project Plan (QAPP). Prior to conducting the audit, a copy of the Final Work Plan, FSP, and QAPP will be reviewed by the auditor (QC Officer or designee). During the TSA the sampling team's adherence to these guidelines will be verified and any deficiencies from the guidelines will be documented. The effect of the deficiencies will be noted, and any necessary corrective actions will be instituted.

Prior to conducting the audit, the auditor will contact the Deputy Project Manager to discuss the audit. This will ensure that the sampling team is properly prepared for the sampling event.

A. Conducting the TSA

The following procedures will be used to conduct the TSA:

1) The auditor will bring the following equipment/documents into the field:

- Copy of the Final Work Plan, FSP, and QAPP, and any relevant memos, correspondence or addenda
- Field laptop
- TSA audit checklist
- Digital camera

2) The following aspects of the sampling event will be audited:

- QA/QC samples
- Sampling methodologies
- Field documentation, including photographs
- Sample management tasks
- Decontamination procedures

B. Corrective Action in the Field

Besides observing and reporting, the auditor is responsible for initiating steps for the start-up of corrective action procedures.

If the auditor witnesses discrepancies in the field between the Final Work Plan, FSP, and QAPP and the performance of the sampling team, the auditor has several options available for corrective action. These options are dependent upon the type of deficiencies observed.

Deficiencies observed and the corrective action taken must be documented in the auditor's log book.

- Minor Deficiencies

  Minor deficiencies are problems where the impact, if any, to the data can be easily eliminated and the deficiency can be corrected or the procedure repeated to achieve the desired result. Minor deficiencies that are observed by the auditor will immediately be brought to the attention of the field team. The auditor and the field team will discuss the problem and agree upon what corrective action is necessary. This will allow for the deficiencies to be corrected immediately in the field.

- Major Deficiencies

  Major deficiencies are events or procedures that substantially deviate from approved work plans, will result in increased project costs not previously approved, or will significantly impact the quality of the data.

  Upon witnessing a major deficiency, the auditor will temporarily stop all related site work and will inform the field team of the problem. The auditor and field team will discuss the deficiency as well as what steps are necessary for corrective action. If the deficiency can be corrected in the field, the auditor may allow work to resume as long as all necessary corrective actions are taken. Information regarding the nature of the deficiency as well as the corrective action(s) taken will immediately be transmitted to the USACE PM, the Malcolm Pirnie Project Manager, and the Deputy Project Manager.

If the deficiency cannot be corrected in the field, a Stop-Work Order will be issued until appropriate measures can be taken to correct the problem. A written report of the major deficiencies will be prepared by the Site QC Officer and submitted to the USACE PM, the Malcolm Pirnie Project Manager, and the Deputy Project Manager. The Stop-Work Order will remain in effect until the proper corrective action(s) can be implemented.

C. Preparation of a TSA Report

The TSA report provides a means of relaying the events of a sampling episode to key personnel. These events could possibly affect the sample integrity (QA/QC) and therefore,

are important to the decisions made regarding analytical data. This report will identify any deficiencies found in the field and will outline the corrective actions that were recommended/implemented to address any minor deficiencies observed. The field audit report will also recommend appropriate corrective actions for any major deficiency noted. Follow-up reports describing completed corrective actions which addressed major deficiencies will be submitted by the Project Manager to the USACE PM.

A quality control field audit report will usually contain the following information:

- Date and location of field audit
- Sample matrices witnessed
- Name of personnel conducting the sampling
- Summary of sample methodology
- Description of any infractions that occurred and the corrective actions taken
- Conclusions
- Recommendations
- Quality control field audit checklist

QUALITY CONTROL FIELD AUDIT REPORT

SUMMARY INFORMATION

1. PROJECT NAME: _____

2. PROJECT ADDRESS: _____

_____

_____

3. PRELIMINARY ASSESSMENT _____ RI/FS _____ RD _____ CONSTRUCTION _____

OTHER _____

4. DATE(S) OF QC FIELD AUDIT _____

5. AUDITOR'S NAME _____ PHONE _____

6. FACILITY CONTACT _____ PHONE _____

7. CONTRACTOR CONTACT _____ PHONE _____

8. PERSONNEL ON-SITE

NAME	REPRESENTING	PHONE

9. AUDITOR'S COMMENTS

_____

_____

_____

_____

10. WEATHER CONDITIONS

SUNNY ; PARTLY SUNNY ; PARTLY CLOUDY ; CLOUDY ; RAIN ; DRIZZLE ; SNOW ; SLEET

TEMPERATURE_____ WIND SPEED_____ WIND DIRECTION_____

11. LEVEL OF PERSONNEL PROTECTION REQUIRED IN WORK PLAN

A B C D

LEVEL OF PERSONNEL PROTECTION ACTUALLY DONNED:

A B C D

12. FIELD SURVEY EQUIPMENT

INSTRUMENT	MODEL	CALIBRATION CHECK	CALIBRATION STANDARD	SPAN SETTING
CONDUCTIVITY METER				
DISSOLVED O_2 METER				
PH METER				
COMBUSTIBLE GAS INDICATOR (LEL/O_2)				
FLAME IONIZATION DETECTOR (OVA)				
PHOTOIONIZATION DETECTOR (HNU)				
TOTAL GAS INDICATOR (CO,H_2S)				
OTHER				

OBSERVATIONS _____

_____

_____

13. DID THE SAMPLING TEAM TAKE PERIODIC SURVEYS OF THE AMBIENT AIR CONDITIONS?

YES NO N/A

14. DID THE SAMPLING TEAM PROVIDE A DECON ZONE DESIGNATING CLEAN AND CONTAMINATED AREAS?

YES NO N/A

15. WERE PHOTOGRAPHS TAKEN? YES NO

16. AUDITOR'S COMMENTS

_____

_____

_____

MONITORING WELL SAMPLING SETUP AND EVACUATION

EVACUATION PROCEDURES

1. WELL CASING CONSTRUCTION STAINLESS STEEL TEFLON PVC OTHER _____

2. DIAMETER OF WELL CASING 2" 4" 6" OTHER _____

3. LOCKING CAPS ON THE WELLS? YES NO N/A PROTECTIVE CASING? YES NO N/A

4. METHOD UTILIZED TO DETERMINE THE STATIC WATER LEVEL

WATER LEVEL INDICATOR OTHER _____

5. REFERENCE POINT THAT THE STATIC WATER LEVEL WAS MEASURED FROM:

SURVEY POINT	TOP OF INNER CASING	TOP OF PROTECTIVE CASING	HEIGHT OF CASING ABOVE GROUND SURFACE

6. WAS THE WATER LEVEL INDICATOR DECONTAMINATED ACCORDING TO STANDARD PROCEDURES BETWEEN EACH WELL?
YES NO N/A

IF NO, METHOD USED: _____

_____

_____

7. EVACUATION METHOD:

BAILER CENTRIFUGAL PUMP PERISTALTIC PUMP BLADDER PUMP SUBMERSIBLE PUMP

GAS DISPLACEMENT PUMP GAS LIFT PUMP OTHER _____

8. TYPE OF HOSE UTILIZED:

POLYETHYLENE TEFLON SILASTIC N/A OTHER _____

9. WAS THE HOSE DEDICATED TO EACH WELL LOCATION? YES NO N/A

IF NO, METHOD OF DECONTAMINATION _____

10. WAS THE PUMP DEDICATED TO EACH WELL LOCATION? YES NO N/A

11. WAS THE PUMP: LABORATORY DECONTAMINATED? FIELD DECONTAMINATED? N/A

12. WAS THE PUMP DECONTAMINATED ACCORDING TO STANDARD PROCEDURES?

YES NO IF NO, METHOD OF DECONTAMINATION _____

13. WAS THE PUMP HEAD OR END OF HOSE WITHIN 6 FEET OF THE DYNAMIC WATER LEVEL DURING EVACUATION?
YES NO N/A

14. WAS THE DECONTAMINATION AREA LOCATED AWAY FROM THE SOURCE OF CONTAMINATION?

YES NO N/A

15. AUDITOR'S COMMENTS

_____

_____

_____

AQUEOUS SAMPLING PROCEDURES

1. AQUEOUS MATRIX SAMPLED:

POTABLE WELL GROUND WATER SURFACE WATER LEACHATE RUNOFF STORM SEWER

SANITARY SEWER OTHER _____

2. TYPE OF SAMPLE: GRAB COMPOSITE IF COMPOSITE - SAMPLES/COMPOSITE _____

3. WAS THE VOA SAMPLE COLLECTED FIRST? YES NO N/A

4. TYPE OF SAMPLING EQUIPMENT:

	MATERIAL OF CONSTRUCTION			
	STAINLESS STEEL	TEFLON	GLASS	OTHER
BAILER	_____	_____	_____	_____
BLADDER PUMP	_____	_____	_____	_____
SAMPLER	_____	_____	_____	_____
COLIWASA	_____	_____	_____	_____
KEMMERER DEPTH SAMPLER	_____	_____	_____	_____
WHEATON DIP SAMPLER	_____	_____	_____	_____
TUB SAMPLER	_____	_____	_____	_____
BACON BOMB	_____	_____	_____	_____

5. TYPE OF LEADER LINE THAT COMES IN CONTACT WITH THE WELL WATER:

TEFLON TEFLON COATED STAINLESS STEEL N/A OTHER _____

6. LENGTH OF THE LEADER LINE _____

7. WAS THE SAMPLING EQUIPMENT DEDICATED? YES _____ NO _____

8. WAS THE SAMPLING EQUIPMENT: LAB DECONTAMINATED? FIELD DECONTAMINATED?

9. WAS THE SAMPLING EQUIPMENT DECONTAMINATED ACCORDING TO STANDARD PROCEDURES?

YES NO IF NO, METHOD OF DECONTAMINATION: _____

10. WAS THE DECONTAMINATION AREA LOCATED AWAY FROM THE SOURCE OF CONTAMINATION?

YES NO

11. ARE DISPOSABLE GLOVES WORN AND CHANGED BETWEEN EACH SAMPLE LOCATION? YES NO

12. AUDITOR'S COMMENTS:

_____

_____

_____

NON-AQUEOUS SAMPLE INFORMATION

1. NON-AQUEOUS MATRIX SAMPLED:

SOIL SEDIMENT SLUDGE CHEMICAL SOLIDS WASTE PILE

OTHER _____

2. TYPE OF SAMPLE: GRAB COMPOSITE IF COMPOSITE - SAMPLES/COMPOSITE _____

3. WAS THE VOA SAMPLE COLLECTED FIRST FROM A DISCRETE LOCATION PRIOR TO HOMOGENIZATION?

YES NO N/A

4. WAS THE SAMPLE HOMOGENIZED PRIOR TO ACQUISITION INTO THE SAMPLE CONTAINERS? YESNO

5. TYPE OF SAMPLING EQUIPMENT:

	MATERIAL OF CONSTRUCTION			
	STAINLESS STEEL	TEFLON	GLASS	OTHER
SPOON/SPATULA	_____	_____	_____	_____
TROWEL/SCOOP	_____	_____	_____	_____
BUCKET AUGER	_____	_____	_____	_____
SPLIT SPOON	_____	_____	_____	_____
SHELBY TUBE	_____	_____	_____	_____
TRIER	_____	_____	_____	_____
PONAR DREDGE	_____	_____	_____	_____

6. WAS THE DRILL RIG, AUGER FLIGHTS, RODS, ETC. DECONTAMINATED ACCORDING TO STANDARD PROCEDURES BETWEEN EACH SAMPLE LOCATION? YES NO N/A

IF NO, METHOD OF DECONTAMINATION _____

7. IF MUD ROTARY DRILLING WAS UTILIZED WHAT WAS THE SOURCE OF THE WATER? _____

8. WAS THE SAMPLING EQUIPMENT DEDICATED? YES_____ NO _____

9. WAS THE SAMPLING EQUIPMENT: LAB DECONTAMINATED? FIELD DECONTAMINATED?

10. WAS THE SAMPLING EQUIPMENT DECONTAMINATED ACCORDING TO STANDARD PROCEDURES?

YES NO IF NO, METHOD OF DECONTAMINATION: _____

11. WAS THE DECONTAMINATION AREA LOCATED AWAY FROM THE SOURCE OF CONTAMINATION? YES NO N/A

12. ARE DISPOSABLE GLOVES WORN AND CHANGED BETWEEN EACH SAMPLE LOCATION? YES NO N/A

13. AUDITOR'S COMMENTS

_____

_____

_____

QA/QC INFORMATION

1. LABORATORY:

NAME _____ PHONE _____

CONTACT PERSON _____

CLP CLP CAPABLE CERTIFIED OTHER _____

3. SAMPLE INFORMATION:

MATRIX	PARAMETER	PRESERVATIVE	CONTAINER DESCRIPTION

3. WHAT ORDER BY ANALYTICAL PARAMETER ARE SAMPLES COLLECTED: _____

_____

4. FIELD BLANKS: YES NO N/A FREQUENCY _____

METHOD: _____

WAS IDENTICAL BOTTLE TO BOTTLE TRANSFER OF WATER UTILIZED? YES NO

5. TRIP BLANKS: YES NO N/A FREQUENCY _____

6. WHAT WAS THE SOURCE OF THE BLANK WATER? LABORATORY DEMONSTRATED ANALYTE-FREE
OTHER _____

7. SAMPLE PACKAGING AND HANDLING:

SAMPLE CONTAINERS LABELED	YES	NO	N/A
COC FORMS COMPLETED	YES	NO	N/A
CUSTODY SEALS	YES	NO	N/A
SAMPLES PRESERVED TO $4^{B}C$:	YES	NO	N/A

8. AUDITOR'S COMMENTS

_____

_____

_____

_____

Attachment 11

Field Modification Form

FIELD MODIFICATION FORM
LOWER PASSAIC RIVER RESTORATION PROJECT

Date:

Document:

Activity:

Requested Modification:

Rationale:

Attachments:

Malcolm Pirnie Project Manager:_____

Malcolm Pirnie Deputy Project Manager: _____

Malcolm Pirnie Site QC Officer:_____

Attachment 12

NJDEP TPH Method

New Jersey Department of Environmental Protection
Office of Quality Assurance
Analytical Method

Title:
Quantitation of Semi-Volatile Petroleum Products in Water, Soil, Sediment and Sludge

Document #:
OQA-QAM-025-10/91

Date: 2/5/01 Revision: 5

Prepared by: Dr. Michael W. Miller

Location: **Master Sets DEP/OQA**

Cleared for Issue by: Joseph Aiello

1.0 SCOPE OF APPLICATION

1.1 Scope

This method utilizes a gas chromatograph fitted with a flame ionization detector (FID). The following petroleum analyses are included in the method:

1.1.1 Quantitative analysis of environmental samples (water, soil, sediment, and sludge) for residues from commercial petroleum products such as crude oil, diesel fuel, waste oil, fuels oils Nos. 2-6, lubricating oil, processed oils and bunker fuel.

The method determines Total Semi-Volatile Petroleum Products (TPHC), also known as Total Petroleum Hydrocarbons. TPHC includes paraffinic, naphthenic, and polynuclear aromatic hydrocarbons (PAHs).

The method must not be used for gasoline contaminated sites.

1.1.2 **Fingerprint** - Identification of unknown petroleum products by comparison of their chromatograms with chromatograms of known petroleum product profiles. Products that can be identified include diesel fuel, fuel oils Nos. 2-6, lubricating oils, bunker fuel and processed oils (2).

1.2 Applicable Program

Underground Storage Tanks (UST), New Jersey Spill Fund, Comprehensive Environmental Response Compensation and Liability Act (CERCLA), Industrial Site Recovery Act (ISRA), Sludge Residuals, and Resource Conservation and Recovery Act (RCRA).

1.3 Method Advantages

1.3.1 The method replaces the TPHC method based on Freon 113 extraction and analysis by infrared spectroscopy.

1.3.2 The FID response produces a TPHC chromatogram that can be used to identify the type of petroleum product present by matching the chromatogram of the unknown sample with the chromatograms of known petroleum products.

1.4 Method Limitations

1.4.1 Quantitative Studies

1.4.2.1 The TPHC is quantitatively restricted to the semi-volatile components since partial loss of volatiles (b.p. < 60°C) occurs during the extraction and/or concentration process.

1.4.2.2 The gas chromatographic conditions are not designed for compounds with carbon numbers greater than C40.

1.4.2.3 Benzene and hydrocarbons that elute from the column before heptane co-elute with the extraction solvent methylene chloride.

1.4.2 Identification Studies

1.4.1.1 The method is most successful with fresh spills of petroleum products. Weathering changes the chromatographic profile. Weathering can be simulated in the laboratory.

1.4.1.2 Absorption, adsorption, biological reactions, and chemical reactions occur in soil, changing the chromatographic profile which reduces the ability to make positive identifications. Method OQA-QAM-018 can be used for more detailed product analysis.

1.5 Matrix

1.5.1 Surface water, ground water, and wastewater.

1.5.2 Soil, sediments or high solids sludge (>50%).

2.0 SUMMARY OF METHOD

2.1 This quantitative TPHC method is adopted from the "Leaking Underground Fuel Tanks Field Manual" of the California State Water Resources Control Board (3). This method is also derived from "Test Methods for Evaluating Solid Waste" USEPA Method 8015B and the Florida Department of Environmental Protection, "Method for the Determination of Total Petroleum Range Organics"(4, 25).

2.2 This petroleum method is adapted with modifications from ASTM Method D3328-82, and the US Coast Guard Oil Spill Identification Procedure for Total Petroleum (1,2).

2.3 Petroleum residues are extracted from sample matrices with methylene chloride. Surrogate compounds are added to all samples before extraction. The methylene chloride extract is analyzed with a gas chromatograph fitted with a capillary column attached to a FID.

2.3.1 The TPHC is determined by integration of the FID chromatogram. Calibration of the gas chromatograph is done with a hydrocarbon standard C8-C40 or where justified and documented, based on historic site-specific data, a standard reference petroleum product.

2.3.2 Identification of unknown residues may be done by comparing their chromatograms with chromatograms of known petroleum products. Samples from old spills are compared to synthetically weathered samples if available. Identification is established when chromatograms match. Method QAM-018 must be used for detailed pattern recognition (6).

2.3.3 The sensitivity of the method is dependent on the level of interference rather than on instrumental limitations. The method detection limit (MDL) for TPHC in soil is approximately 10 ug/g and in water 30 ug/L. These MDLs can be achieved without concentrating the extract.

2.3.4 Dynamic Range

2.3.4.1 TPHC

Soil 30-1000 ug/g
Aqueous 0.1-500 mg/L

2.3.4.2 Individual Compounds

Soil 1.0-500 ug/g
Aqueous 0.2-200 ug/L

3.0 INTERFERENCES

3.1 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with tap water, distilled water, methanol, and methylene chloride.

3.2 High purity reagents such as Burdick and Jackson GC2 methylene chloride, Baker capillary grade methylene chloride or equivalent must be used to minimize interference problems.

3.3 Before processing any sample, the analyst should demonstrate daily, through the analysis of a method blank, that the entire system is interference-free.

3.4 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from source to source (e.g. fatty acids, biogenic materials, oxidized biodegradation products), depending upon the nature and diversity of the site being sampled. The cleanup procedure, EPA Method 3630B can be use to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the method detection limit (MDL) (4).

3.5 Naturally occurring alkanes may be detected by this method and may interfere with product identification. Naturally occuring plant waxes include odd carbon number alkanes from n-C_{25} through n-C_{35} and exhbit a dominant odd/even chain length distribution. Leaf hydrocarbons also may be detected.

3.6 A vial septum should be penetrated and extracted with methylene chloride to evaluate the potential alkane distribution that could occur in re-analyzed extracts. Vial septums should be changed after each analysis.

3.7 Particulates interfer with the determination of disolved petrolem products in ground water. Petroleum products adsorb on the surface of particulates. The groundwater samples should be filtered through glass fiber filters to determine dissolved TPH.

4.0 SAFETY

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been defined precisely. Each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) should also be made available to all personnel involved in the chemical analysis. Additional reference to laboratory safety are available and have been identified for use by the analyst (8,9).

5.0 APPARATUS AND EQUIPMENT

5.1 Sampling Containers

5.1.1 Prior to use, wash bottles and cap liners with aqueous detergent solutions and rinse with tap water, distilled water, and methylene chloride. Allow the bottles and containers to air dry at room temperature, place in a 150°C oven for one hour, then remove and allow to cool in an area known to be free of organic analytes.

5.1.2 Screw cap bottle - 40 mL PTFE-faced silicone cap liners.

5.1.3 Narrow mouth bottles - 1 liter, amber, PTFE faced silicone cap liners.

5.1.4 Wide-mouth glass jar-four ounce, amber, PTFE faced silicone cap liners

5.2 Glassware

5.2.1 Serum bottles - 100 mL, 10 mL, 2 mL crimp-top, PTFE-faced silicone cap liners.

5.2.2 Pasteur pipets

5.2.3 Screw-cap Erlenmeyer flasks - 250 mL, with PTFE faced silicone cap liners.

5.2.4 Volumetric flasks - 10 mL, 25 mL, 100 mL

5.2.5 Kuderna-Danish apparatus (KD)

5.2.6 Sepratory funnels - 2 L Pyrex, Tefon stopcoat

5.2.7 Soxhlet Extractor

5.3 Apparatus

5.3.1 Rotary shaker table, 350 rpm minimum

5.3.2 Analytical balance capable of accurately weighing 0.0001 g.

5.3.3 A gas chromatograph with split/splitless injector, equipped with a capillary column, capable of temperature programming.

5.3.3.1 Column-30m long x .53mm ID, .5um film thickness dimethyl polysiloxane coating (Restek, J&W Scientific or equivalent). This column will allow for the resolution of alkanes from nC8 to nC40, as well as the resolution of phytane/nCl8 and pristane/nCl7. This column will also allow for the resolution of the petroleum products listed in Section 1.1 (21). Low bleed columns must be used. Equivalent columns maybe used.

5.3.3.2 Column - 30m long x 0.32mm ID, 0.25um film thickness, 95% dimethyl-5% diphenyl polysilxane (J&W Scientific, Restek or equivalent). This column has simillar resolution to 5.3.3.1 but high bp compounds elute later. Equivalent columns maybe used.

5.3.3.2 Column - 100m long x .25mm ID., 0.5um film thickness, capillary, Protocol DH (Supelco or equivalent (22).

5.3.3.3 Detector - Flame Ionization Detector (TPHC only)

5.3.4 An autosampler is recommended.

5.3.5 Boiling chips - Solvent extracted approximately 10/40 mesh.

5.3.6 Water bath - Top, with concentric ring cover, capable of temperature control. The bath should be used in a hood.

5.3.7 Gas-tight syringe - One milliliter (mL) with chromatographic needles.

5.3.8 Microsyringes – l0uL, 100uL, 200uL

5.3.9 Continuous liquid-liquid extraction apparatus.

5.3.10 Liquid chromatographic column - 400 x 20mm with course frit, teflon stopcock.

5.3.11 Magnetic stirrer and 2-inch Teflon coated stirring bars.

5.3.12 Nitrogen concentration system composed of a precleaned pasteur pipet, with a small plug of glass wool loaded at the tip end, and filled with approximately 1-2 cm of precleaned alumina. The top of the pipet is attached to a hydrocarbon free nitrogen gas source using precleaned Teflon tubing. This concentration step should be performed at room temperature or lower to retain light end compounds.

6.0 REAGENTS

6.1 Purity of Reagents - Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee and Analytical Reagents of the American Chemical Society.

6.2 Reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest. (ASTM Specification D1193, Type ii).

6.3 Methylene chloride, methanol, carbon, disulfide and hexane - pesticide grade, Burdick and Jackson GC2, Baker Capillary Grade or equivalent.

6.4 Sodium sulfate - (ACS) granular, anhydrous. Purify by heating at 400°C for four hours in a shallow tray, cool in a desiccator and store in a sealed glass bottle.

6.5 Silica gel - Grade 923 (100/200) desiccant. Before use, activate for at least 16 hours at 130°C in a shallow glass tray that is loosely covered in foil. Cool and store as in Section 6.4.

6.6 Silica granular, fine (60-120 mesh) Fisher 5151-10. Purify by Soxhelt extraction with methylene chloride for four hours. Dry at 80°C. Store in glass bottle.

6.7 Hydrochloric acid, 1:1 - Mix equal volumes of (ACS) concentrated HCl and distilled water.

6.8 Stock Standards

6.8.1 Normal Paraffinic Hydrocarbons - Prepare a Methylene chloride solution containing a mixture of components ranging from octane to tetracontane

(nC8-nC40), or selected individual normal paraffins (2 mg/ml ea). The standard must include a minimum of 12 compounds including C8, C18, C20, C32, and C40. For product identification purposes the mixture also may contain C17, pristane and phytane (2 mg/ml ea.) . A separate pristane, phytane, C17, and C18 column resolution standard may be used. An appropriate dilution of the mixture is analyzed under normal analytical conditions to determine retention times and the Relative Response Factors for the Compounds. A 10% carbon disulfide/ 90% methlyene chloride solvent mixture may be used if standard do not remain in solution. (Mixtures are available from Supelco, Restek (31266), NSI Solutions and Ultrex).

6.8.2 Diesel and Fuel Oil Profile Standards - Where applicable prepare commercial diesel, waste motor oil and/or fuel standards in methylene chloride. Weigh 300mg oil to the nearest 0.1 mg, into a 10 mL volumetric flask. Dilute to volume with methylene chloride (30 mg/ml). Standards are available from Restek Inc., Bellefonte, PA.

6.8.3 Surrogates (SS) and Internal Standards (IS) - The surrogates (chlorobenzene, ortho-terphenyl [OTP] and tetracosane-d50), and internal standard (5-androstane) are prepared separately by carefully weighing 100 mg of each compound in a 100 mL volumetric flask. Dilute to volume with methylene chloride. The final concentration of each compound is one ug/ul. The laboratory is free to choose any two or more surrogates that cover the carbon range (C8 - C40). However chlorobenzene is required. Standards are available from Restek Inc.

6.8.4 Quality Control Check Standard (Blank Spike) - The QC check standard concentrate (30 mg/ml) of fuel oil #2 must be prepared by the laboratory using stock standards prepared independently from those used for calibration (Section 6.8.2). If the contamination at the site is known, other fuel standards can be used. Standards are available from Supelco, Restek or Ultrex.

6.8.5 Matrix spiking standard (MSS) - Prepare the MSS in methylene chloride from a Fuel oil #2 standard in Section 6.8.2 or another oil for site speific work . The laboratory may use the hydrocarbon calibration standard noted in section 6.8.1 as the MSS for unknown petroleum sources.

7.0 CALIBRATION

7.1 Initial Calibration

7.1.1 Rentention time windows

a. Before establishing windows, make sure the GC system is within optimum operating conditions. Serial injections over less than a 72 hr period result in retention time windows that are too tight.

b. Calculate the mean and the standard deviation of the three retention times (use any function of retention time; including absolute retention time, or relative retention time) for each surrogate.

c. Plus or minus three times the standard deviation of the mean retention times for each surrogate will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. The default value for the retetion time shall be a minimum of +/- 0.05 minutes, if the standard deviation is zero.

d. Establish the midpoint of the retention time window for each surrogate by using the absolute retention for each surrogate from the mid-concentration standard of the initial calibration. The absolute retention time window equals the midpoint $\pm$ 3 SD, where the standard deviation is determined as described in Section b.

e. The laboratory must calculate retention time windows for each surrogate on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

7.1.2 FID Internal Standard Calibration for Quantitation of TPHC - Calibrate the GC-FID with an initial fivepoint, (i.e., concentration of individual components 1 ng/uL, 5ng/uL, 10ng/ul, 20 ng/uL, 50ng/uL), Total Petroleum Hydrocarbons calibration curve (Section 6.8.1). The lowest concentration point in the calibration curve should be near the MDL. The highest concentration point should be twice the expected sample concentration and within the linear instrument range. To maintain the standards in solution, a 10% carbon disulfide/90% methylene chloride solvent may be required. Prepare the calibration standard to contain 5.0 ng/ul of surrogate and GC internal standard. The relative standard deviation (RSD) for the surrogates and each calibration compound's relative response factor (RRF) must be less than or equal to 20%. If not, the calibration curve must be used to determine concentration. A minimum correlation coeffient of 0.95 is required. Also calculate the response for total petroleum hydrocarbons (TPHC) which is the average of the RRFs for the nC8-nC40 compounds (Section 11.2.2). The RSD must be less than 20%. The chromatograms should be checked for mass discrimination (Section 7.3). Standards with concentrations greater than 20 mg/L may need to be equilibrated to room temperature prior to analysis.

7.1.3 TPHC External Standard Calibration - The laboratory has the option of using an external calibration procedure. Prepare the standards as directed in Section 7.1.2 omitting the internal standard. Calculate response factors (RF) instead of relative response factors for each compound and the surrogates. The relative standard deviation (RSD) of each calibration compound's response factor (RF) must be less than 20%. If not the calibration curve must be used to determine concentration. A minimum correlation coefficient of 0.95 is required. Also

calculate the response for total petroleum hydrocarbons (TPHC) which is the average of the RFs for the nC8-nC40 compounds (Section 11.3).

7.1.4 TPHC Petroleum Product Calibration - If the laboratory has a site specific project and the petroleum product contamnation is known the GC_FID maybe calibrated using a reference petroleum product (Section 6.8.2). Calibrate the GC-FID with an initial five point, (i.e. concentration total petroleum 0.1 mg/ml, 0.2mg/ml, 1.0mg/ml, 2.0mg/ml, 5.0mg/ml) calibration curve. Calculate the response factor (RF) for TPHC (Section 11.2). The RSD of the TPHC responce factor must be less than 20%. If not the calibration curve must be used to detirmine concentration. A minimum correlation coefficient of 0.95 is required.

7.1.5 Petroleum Profile Calibration - If the method is used only for qualitative petroleum product profiling, a single point GC-FID retention time calibration is required. Prepare a 2 mg/ml standard from the products in section 6.8.2 (23). Prepare a 20 ng/ul carbon number standard from section 7.12.

7.2 Daily Calibration

7.2.1 The resolution check is required for ~~fingerprint determination~~. The resolution of the chromatographic column should be checked daily. Inject two uL of the calibration mixture (10 ng/ul) (section 7.1.2) into the chromatograph. Visually inspect the chromatograms for separation between phytane and nC18.

If a standard fuel oil #2 is used as the calibration mixture, the resolution of the chromatograph column can be monitored by visually checking the resolution of the pair, nC17-Pristane and nC18-phytane. These compounds occur in all fuel oil #2 products.

7.2.2 The working calibration curves or calibration factors for TPHC, must be verified on each working day and after every 12 hours by the measurement of one or more calibration standards, (10 ng/ul). Calculate the percent difference (D%) between the verified RF/RRF and the inital responses RF/RRF for TPHC and the surrogates. The %D must not exceed 20%. If the %D exceeds 20% the instrument must be recalibrated. Reanalysis of all samples analyzed after a non compliant standard is required.

7.2.3 The retention times of surrogates in the calibration verification standard analyzed at the beginning of the analytical shift must fall within the absolute retention time windows calculated in Sec. 7.1.1b. The purpose of this check is to ensure that retention times do not continually drift further from those used to established the widths of the retention time windows. If the retention time of any surrogate at the beginning of the analytical shift does not fall within the ± 3 SD

window (minimum +/- 0.05 min.), then a new initial calibration is necessary unless system maintenance (Sec. 7.11) corrects the problem.

In addition, the retention times of all surrogates in the subsequent calibration verification standards analyzed during the analytical shift must fall within the absolute retention time windows established in Sec. 7.1.1d.

7.2.4 Surrogate Standards (SS), GC Internal Standard (IS) - The SS and IS responses and retention times in the calibration check standard must be evaluated during or immediately after data acquisition. If the retention time(s) for the SS or IS is outside the determined RT window, the chromatographic system must be inspected for malfunctions and corrections must be made. If the area(s) for the SS or IS changes by +/- 50% from the last daily calibration standard check, the GC must be inspected for malfunctions and corrections must be made.

7.3 Mass Discrimination

7.3.1 Mass discrimination can take place in the injection port. The heavier molecules do not enter the column as a defined plug of vapor with the lighter molecules.

7.3.2 Mass discrimination is minimized by placing a small plug of silanized glass wool one centimeter from the base of the glass injection liner. The end of the capillary column is placed just below the glass wool.

7.3.4 The calibration chromatogram (Section 7.1.1) is inspected for mass discrimination. The response factor ratio C32/C20 should be greater than 0.8. If the ratio is less, reposition the column in the glass liner until mass discrimination is minimized.

8.0 QUALITY CONTROL

8.1 Each laboratory that uses this method is required to operate a formal quality control program which conforms with New Jersey Regulation N.J.A.C. 7:18-4.7 (13). The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of QC samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with laboratory established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst must make an initial, one-time demonstration of the ability to generate acceptable accuracy and precision with this method (Section 8.2).

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted to improve the separations by changing the GC conditions or column. Each time such a modification is made to the method, the analyst is required to repeat and document the procedure in Section 8.2.

8.1.3 Each day before calibration and after the calibration, the analyst should analyze a reagent blank (instrument blank) to demonstrate that interferences from the analytical system are under control.

8.1.4 With each sample batch, the analyst must analyze a method blank to demonstrate that interferences from sample extraction are under control. Target compounds' concentrations in the blank should be no more than 5x MDL. If blank levels for any component are above 5x MDL and the sample concentrations present in the samples are greater than 10X then the samples may be quantified and qualified. If the blank concentration is greater than 5X MDL and the sample concentrations present in the samples are less than 10 X the blank level, the affected samples should be re-extracted and re-analyzed. If a sample cannot be re-extracted or re-analyzed, the data should be qualified as such. Samples should not be blank corrected.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.3. The frequency of the check standard analyses is equivalent to 5% of all samples analyzed.

8.1.6 The laboratory must spike all samples with the surrogates to monitor recovery. This procedure is described in Section 8.4.

8.1.7 The laboratory must spike a minimum of five percent or one per batch, which ever is more frequent of all samples in each matrix, with the MSS (Section 6.8.5) to monitor and evaluate laboratory data quality. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable precision and accuracy, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing fuel oil #2 at a concentration of 30 mg/ml in methylene chloride (Section 6.8.4). The QC check sample concentrates must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Aqueous

Analyze four 1 L aliquots (TPHC) of the well-mixed reagent water spiked with 0.5 mL QC check sample concentrate and 20 uL of each surrogate according to the method beginning in Section 10.1.

8.2.3 Soil and Sediment

a. Prepare a TPHC QC check sample to contain 200 ug/g of a reference oil (6.8.2) by adding 0.2 mL of QC standard concentrate to 30g of reagent sodium sulfate (6.4). Also add the appropriate surrogates.

b. Analyze four aliquots of silica spiked with QC check standard concentrate according to the method beginning in section 10.3.

8.2.4 For each matrix calculate the average recovery (X) and the standard deviation of the recovery (s) for #2 fuel, using the four results. The average percent recovery should be between 70-120%. samples. The laboratory is to establish the criteria for the standard deviations as described in USEPA Method 8000 A (4).

8.2.5 The FID retention times of the surrogates and internal standards must match the calibration standard as described in 7.2.3.

8.3 The laboratory must analyze a Total Petroleum Hydrocarbon (TPHC) QC check sample with every 5% of the samples. The check sample shall be prepared as directed in Section 8.2.1. The recovery of TPHC shall be in ranges specified in Section 8.2. The FID retention times of the surrogates and internal standards must match the previous calibration as described in 7.2.3.

8.4 As a quality control check, the laboratory must spike all samples with the surrogates chosen in Section 6.8.3 and calculate the percent recovery (P) of the Surrogate based on the FID response.

$$\%P = \frac{A_s}{A_s} \times 100$$

A_s = Area response of SS or IS in check sample

A_s = Average area response of SS or IS in standard

8.4.1 For the surrogate standards, the laboratory must develop separate accuracy statements of laboratory performance for each matrix. An accuracy statement for the method is defined as Percent Recovery $\pm$ Standard Deviation ($P \pm s$). The accuracy statement should be developed by the analysis of four aliquots as described in Section 8.2, followed by the calculation of P and s. Alternatively, the

analyst may use four data points gathered through the requirement for continuing quality control in Section 8.3. The accuracy statements should be updated regularly. The initial recovery may be set 60 to 120 percent.

8.4.2 Calculate upper and lower control limits for %P for the surrogate standard in each matrix.

Upper Control Limit (UCL) = P +3s
Lower Control Limit (LCL) = P - 3s

The UCL and LCL can be used to construct control charts that are useful in observing trends in performance (14).

8.4.3 The following corrective actions can be taken when the percent recovery of chlorobenzene, androstane and/or tetracosane-d50 are outside of the recovery range:

8.4.3.1 Check calculations to assure there are no errors.

8.4.3.2 Check instrument performance, check the sample preparation procedure for loses due to temperture control and internal standard and surrogate solutions for degradation contamination, etc.

8.4.3.3 Reanalyze the sample or extract if the steps above fail to reveal a problem. If reanalysis yields surrogate recoveries within the stated limites, the reanaysis data should be used.

8.4.3.4 If the surrogate could not be measured because the sample was diluted prior to analysis, qualify the surrogate recovery. If the surrogate co-elutes with a compound (as in coal tar), report only the recovery of the backup surrogate tetracosane-d50. Qualify the out of range surrogate on the data table. No additional corrective action is required.

8.5 Matrix Spike Analysis - The laboratory must, on an ongoing basis, spike (MS) and analyze at least 5% of the samples for each matrix from each sample site being monitored to assess accuracy. The spike should be the matrix spiking standard (MSS) (Section 6.8.5).

8.5.1 Report the recovery as the average of the individual recoveries of the components nC8-nC40, if the hydrocarbon standard is used.

8.5.2 The laboratory should establish there own acceptance criteria for % recovery (R) as in Section 8.2.4. Recoveries of > 70% should be achieved.

8.6 Sample Duplicate - The laboratory must, on an ongoing basis, analyze 5% of the samples for each matrix in duplicate. Both results are to be reported. (No specific criteria concerning the relative precent difference (RPD) exists at this time. However, results should not differ by more than 50%.) The laboratory should establish their own acceptance creteria for RPD based on control charts. A matrix spike duplicate may be used if no postive TPHC samples are in the batch.

8.7 Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

8.8 The laboratory shall determine the method detection limits (MDLs) for the fuels of interest using the methods of 40 CFR 136 Appendix B (7). The MDLs must be confirmed weekly by analyzing a low level standard (2x MDL).,

9.0 SAMPLE COLLECTION PRESERVATION

9.1 Aqueous Matrix

9.1.1 Collect a representative water sample in a 1L narrow mouth bottle. A delay between sampling and analysis of greater than four hours requires sample preservation by the addition of 5ml HCl (Section 6.7). Confirmation of a pH < 2 must be obtained in the field.

9.1.2 Sample must be chilled to 4+/-2°C on the day of collection and stored at 4+/-2°C until analyzed.

9.1.3 Samples must be extracted within seven days from the time of collection or five days from verified sample time of receipt (VSTR). Extracts must be analyzed within 40 days of extraction.

9.2 Solid Matrix

9.2.1 Collect a representative soil-sediment sample in a four-ounce, wide-mouth jar with a minimum of air space.

9.2.2 Samples must be chilled at 4+/-2°C on the day of collection and stored at 4+/-2°C until analyzed.

9.2.3 Samples must be extracted within seven days from the time of collection or five days from VSTR. Extracts must be analyzed within 40 days of extraction.

10.0 PROCEDURES

10.1 Dissolved Product

10.1.1 Sealed Extraction (14)

a. Mark the liquid level on the outside of the one liter bottle. Check the sample for floating product. Measure/adjust pH to 2 with 6N HCl if necessary.

b. Add a two inch teflon stirring bar, 30 ul of surrogate standards (Section 6.8.3), and 30 mL of methylene chloride. Seal the sample bottle.

c. Place the bottle on a magnetic stirrer. Adjust the speed so that there is a vortex. Stir for five minutes and then release any pressure. Continue stirring for 15 minutes.

d. Transfer the methylene chloride extract to a serum bottle for storage using a Pasteur pipet. Add 30 mL of methylene chloride to the sample bottle, seal, return to the magnetic stirrer and stir for twenty minutes. Fill the empty sample bottle to the mark with water. Determine the sample volume in a liter graduated cylinder.

e. Combine the methylene chloride extracts, filter and dry the extract through glass wool and sodium sulfate. Adjust the extract volume to 50 mL in a volumetric flask. Store the extract in a 100 mL crimp top serum bottle at 4°C.

f. Emulsions

1. Emulsions can be broken by centrification

2. Continuous extraction can be used on samples known to form emulsions, USEPA Method 3520 (4).

g. Screen the extract before concentrating (Section 10.4). Dry and Concentrate the extract as in section 10.1.2.e through 10.1.2i.

10.1.2 Separatory Funnel Extraction (7)

a. Aqueous samples are usually extracted using separatory funnel techniques. If emulsions prevent achieving acceptable solvent recovery

with separatory funnel extraction, continuous extraction (Section 10.1.2) may be used. The separatory funnel extraction scheme described below assumes a sample volume of 1 L. When a sample volume of 2 L is to be extracted, use 250, 100 and 100-mL volumes of methylene chloride for the serial extraction.

b. Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2 L separatory funnel. Measure/adjust pH to 2 with 6N HCL. Pipet 30 uL of the surrogate standard spiking solution into the separatory funnel and mix well (Section 6.8.3).

c. Add 60 mL of methylene chloride to the sample bottle, seal and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min.

If the analyst must employ mechanical techniques to the complete phase separation, the optimum technique depends upon the sample. The techniques may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250 mL Erlenmeyer flask with a glass stopper.

If the emulsion cannot be broken (recovery of less than 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the extract to the chamber of a continuous extractor and proceed as described in Section 10.2.3.

d. Add a second 60 mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. Label the combined extract. Screen the extract (Section 10.4) before concentrating.

e. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D concentrator if the requirements of Section 8.2 are met.

f. Pour the combined extract through a solvent rinsed drying column (Section 5.3.10) containing about 10 cm of anhydrous sodium sulfate, and

collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

g. Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask for each fraction. Prewet each Snyder column by adding about 1 mL of methylene chloride to the top. Position the K-D apparatus in a hot water bath (60°C to 65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of the distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with one to two mL of methylene chloride. A five mL syringe is recommended for this operation.

h. Add another one or two clean boiling chips to the concentrator tube of each fraction and attach a two ball micro Snyder column. Prewet the Snyder column by adding about 0. 5 mL of methylene chloride to the top. Place the KD apparatus on a hot water bath and allow it to drain and cool for at least 10 min. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with approximately 0.2 mL of methylene chloride. Adjust the final volume to 1.0 mL with the solvent. Transfer to a 1 ml GC autosampler vial and add 20 uL of the GC internal standard (5-androstane). If the internal standard method is used.

i. Determine the original sample volume by refilling the sample bottle to the mark with water and transferring the liquid to a 1000 mL graduated cylinder. Record sample volume to the nearest five mL.

j. Other extract concentation methods can be used if the recoveies of the surrogates and the QC check samples meet the requirements of the method.

10.2.3 Continuous Extraction (4)

a. Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Transfer the sample to the continuous

extractor. Measure/adjust pH to 2 with 6N HCl. Using a pipet, add 30uL of surrogate standard spiking solution and mix well. Add 60 mL of methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the extractor.

b. Repeat the sample bottle rinse with an additional 50 to 100 mL portion of methylene chloride and add the rinse to the extractor.

c. Add 200 to 500 mL of methylene chloride to the distilling flask, and sufficient reagent water to ensure proper operation, and extract for 24 hours. Allow to cool, then detach the distilling flask. Screen the extract (Section 10.4) before concentrating. Dry and concentrate the extract as in section 10.1.2.e through 10.1.2.i.

10.3 Sample preparation, soils and sediments (1,12)

10.3.1 Homogenize the soil sample with a solvent-rinsed stainless steel spatula. Weigh about five grams of the sample to +.01g into a tared aluminum pan. Dry at 105 degrees Celsius for 12 hours and calculate the percent solids content (Section 11.3.4).

10.3.2 Soxhlet Extraction - If the samples contain residual oil and tar, the EPA soxhlet extraction method 3540 should be used (2). Method 3540 may be used for all sample types.

a. Quickly blend 10-30g of the solid sample with 10-30g of anhydrous sodium sulfate and place in an extraction thimble. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the Soxhlet extractor is an acceptable alternative for the thimble. Add 30uL of the surrogate standard spiking solution onto the sample. The blending of the soil with the anhydrous sodium sulfate can lead to the loss of volitile components (C8-C12). If the sample is expected of containing a large volitile component such as Jet fuel, this drying process may be omitted.

b. Place 300 mL of the extraction solvent into a 500-mL round-botton flask containing one or two clean boiling chips. Attach the flask to the extractor and extract sample for 16-24 hours at 4-6 cycles/hr.

c. Allow the extract to cool after the extraction is complete. Screen the extract before continuing (Section 10.4). Dry and concentrate the extract as in Section 10.1.2e through 10.12h.

10.3.3 Gyrotory Shaker Extraction

a. Add 30g (+/- .1 g) (wet weight) of the sample with a stainless steel spatula to a 250 mL acid-cleaned, solvent-rinsed, capped Erlenmeyer flask. Mix in 30 g of sodium sulfate. If the sample has excessive moisture, add additional amounts of sodium sulfate. This procedure must be done quickly to reduce evaporation losses.

b. Add 50 mL of methylene chloride, 30 uL of surrogate standard spiking solution, screw on the top, and shake the mixture vigorously for 1 minute, vent the flask (Section 6.8.3).

c. Place the 250 mL flask on a rotary shaker unit, positioned behind a safety shield, in a fume hood. Adjust the agitation rate of the shaker device to 400 RPM and extract the samples under these conditions for exactly 30 minutes.

d. After 30 minutes, the rotary shaker should be turned off and Erlenmeyer flasks removed to a stationary location. The analyst should permit suspended solids and particulate matter in the flasks to settle for approximately 30 minutes.

e. Using a pasteur pipet, carefully transfer the supernatant extract to a 100 mL crimp top serum bottle for storage. Do not agitate the sediment layer.

f. Extract the sample with two additional 30mL volumes of methylene chloride. The sample should be sonicated for 0.5 minutes before the last extraction. This will insure the complete dispersion of the soil. Combine all of the extracts, in the serum bottle for storage. Screen the extract before continuing (Section 10.4). Dry and concentrate the extract as in section 10.1.2e through 10.1.2.h.

10.3.5 Other Extraction Methods

a. Extraction methods listed in SW846 may be used such as 3545 Pressurized Fluid Extraction, 3541 Automated Soxhlet Extraction and 3550 Sonication.

b. The laboratory must conduct a one time demonstration of the ability to

generate acceptable accuracy and precision (Sectin 8.2). The demonstration must include comparison results with real samples that cover the C8-C40 carbon range. The laboratory must meet the sensitivity

requirements of Section 2.3.3, the dynamic range of section 2.3.4, and the surrogate recovers of section 8.4.1.

c. Routine analysis using alternate extraction methods must meet the surrogate reqiurements of section 8.4.1.

10.4 Preliminary Analysis of Extracts (Screening)

10.4.1 Adjust the chromatograph for maximum sensitivity.

10.4.2 Inject 1 uL of the sample extract using an auto sampler.

10.4.3 A complete profile of the extract should be obtained without saturating the detectors. The largest peak should be a minimum of 50% and a maximum of 90% full span. If the response is too high, the extract should be diluted. if the response is too low, concentrate the extract. If the response meets the criteria, determine the extract volume and go to section 10.5 or 10.6.

10.5 Cleanup for Quantitation (4)

10.5.1 Should the sample require additional cleanup due to interfering compounds, polar compounds or a large unresolved hump without destinct peaks, in the chromatogram, use EPA Method 3630 - silica gel cleanup. Combine fraction 1 (pentane; aliphatics) and fraction 2 (methylene chloride/pentane [2: 3 v/v] aromatics) for total petroleum hydrocarbons (TPHC) analysis.

10.6 Chromatographic Analysis

10.6.1 One milliliter of extract ready for analysis should be transferred to a one mL GC auto sampler vial.

10.6.2 All extracts, standards and blanks, should contain the GC internal standard (30 ug/L) when applicable.

10.6.3 Inject one to two uL of extract using an autosampler device or the solvent plug method.

10.7 Recommended Chromatographic Conditions (21)

10.7.1 Temperature Program for RTX-1 Column:

Initial Value	40°C
Initial Time	2.00 min

Program Rate	12.00°C/min
Final Value	320°C
Final Time	10 min
Total Run Time	35 min
Carrier Gas	He

10.7.2 Injection Port Temperature 330°C

10.7.3 Detector Temperature 330°C

10.7.4 Instrument Performance

a. All of the peaks contained in the standard chromatograms must be sharp and symmetrical. Peak tailing must be corrected. If only the compounds eluting before ethylbenzene give random response, have unusually wide peak width, are poorly resolved, or are missing, the problem is usually traceable to the injection port temperature.

b. Check the precision between consecutive QC check samples. A properly operating system should perform with an average relative standard deviation of less than 10%. Poor precision is generally traceable to pneumatic leaks.

c. Monitor the retention time for each analyte using data generated from calibration standards. If individual retention times vary by more than ±3 standard deviations (7.1.1) over a twelve hour period, the source of retention data variance must be corrected before acceptable data can be generated.

d. The instrument sensitivity must be maximized. Injection of 2ul of a 1ng/ul hydrocarbon standard should yield a detector signal-to-noise ratio of between 5:1 and 15:1 for the individual alkanes.

10.8 Analysis Sequence

10.8.1 This method uses a 12hr clock. The time sequence begins with the analysis of the first initial calibration standard. Continuing calibrations must be verified every 12hrs.

10.8.2 Sequence

1. Instrument Blank
2. Initial Calibration

3. Samples (including Method Blanks, and QC samples)
4. Continuing Calibration (every 12hrs.)

11.0 CALCULATIONS

11.1 Concentration of Petroleum Hydrocarbons

11.1.1 To calculate the concentration of total petroleum hydrocarbons in the sample, the area response attributed to the petroleum must first be determined. This area includes all of the resolved peaks and the unresolved "envelope". This total area must be adjusted to remove area response of the internal standards, solvent, surrogates and the GC column bleed.

11.1.2 Establishing the baseline

a. Column bleed is defined as the reproducible baseline shift that occurs during temperature programming of the GC column oven. To determine this area, a methylene chloride blank injection should be analyzed at the beginning of the day and after every 12 hours to determine the baseline response. The baseline is then set at a stable reproducible point just before the solvent peak. This baseline should be extended horizontally to the end of the run. The area for the blank run that must be subtracted from the actual sample run, includes all of the area between C_8 and C_{40}.

Baseline response within ± 20%

b. The baseline for the sample should be set in the same manner. The area in the sample will contain the area attributed to petroleum and that attributable to the baseline. The petroleum area must be calculated by subtracting the baseline area and area for the standards from the total area. The TPHC is then calculated according to the equation in Section 11.1.4.

c. As the concentration of TPHC in the sample approaches the detection limit, the baseline correction becomes more critical.

11.1.3. Mass discrimination must be kept to a minimum by placing a small plug of silanized glass wool one cm from the base of the glass injection liner. The capillary column should be placed just below the glass wool. A full range alkane standard should be run to test the degree a mass discrimination before performing any actual sample analyses. The reponse factor ratio of C_{32}/C_{28} should be greater than 0.8. If less than 0.8, reposition the column in the glass liner until the mass discrimination is minimized.

11.1.4 Concentration based on Internal Standard

RRT version - Initial or CCC

a. From sample analysis, determine the total area (A) of the unknown and calculate the concentration. The analyst must take care when calculating total product areas that the appropriate baseline is used.

(1) Aqueous Matrix

$$C = \frac{(A)\ (C_{is})\ (D)\ (Ve)}{(RRF)\ (A_{is})\ (V_o)\ (Vi)}$$

Where:

C = Concentration of Analyte or concentration of Total Petroleum Products, ug/L

A = Area response of compound or TPH to be measured.

D = Dilution Factor

C_{is} = Concentration of internal standard added to extract

A_{is} = Area response of internal standard

RRF = Relative response factor of analyte (section 11. 2.2). For TPHC RRF is the average of all the nC8-nC40 RRFs.

V_o = Final volume of extract, uL

V_i = Volume of extract injected, uL

V_s = Volume of sample, L

(2) Soil and Sediment

$$C = \frac{(A)\ (C_{is})\ (D)\ \ Ve}{(RRF)\ (S)\ (A_{is})\ Vi}$$

C = Concentration of compound to be measured, or Concentration of TPHC, ug/g

S = Dry sample weight, grams

11.2 The FID Relative Response Factor (RRF) calculation for a specific compound or TPHC.

$$RRF = \frac{C_s \times A_c}{A_s \times C_c}$$

Where:

C_c = Concentration of calibration compound or TPHC standard, ug/ml

C_s = Concentration of internal standard, 5-androstane, ug/ml

A_s = Area Response of internal standard

C_c = Area of calibration compound or TPHC standard

11.3 External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of analyte injected, from the peak response, using the calibration curve or the calibration factor determined in Step 7.1.3. The concentration of a specific analyte is calculated as follows:

(1) Aqueous samples

$$\text{Concentration (ug/L)} = \frac{(A)\,(D)\,(Vc)}{RF\,(V_o)\,(Vi)}$$

RF = Response factor of analyte (Section 7.1.2) or TPHC average response of all th nC8 - nC40 RFs.

(2) Nonaqueous

$$\text{Concentration (ug/g)} = \frac{(A)\,(D)\,(Vc)}{RF\,(s)\,(Vi)}$$

11.4 Matching Chromatographic Patterns - The following provides the basis for identification of the type of petroleum product. Method OQA-QAM-018 should be used to make more definative identifications.

11.4.1 Interpretation (from ASTM D 3328, (1))

a. Basis of Matching - The source of a spilled petroleum oil can be identified by comparison of the chromatogram to that of a suspected source oil or

that of a reference oil. Identical petroleum products give identical chromatographic peak patterns.

b. Chromatogram Features - The major features of the chromatogram that are used for comparison are listed as follows:

A FID chromatogram shows the features of a homologous series of normal paraffins, the isoprenoid hydrocarbons pristine and phytane, the other resolved peaks and the unresolved envelope. All of these features are used to characterize petroleum oil.

c. Weathering effects: - When petroleum is spilled on open water or spilled on open ground, weathering will progress rapidly. A thin slick on open water may lose significant amounts of its components, up to nC15, 271°C, atmospheric boiling point, within 48 hours of being spilled. Soil samples will lose the soluble fraction through leaching. Biological degradation of petroleum begins within seven days. It is important to be aware of the effects of weathering when matching chromatograms of spill samples more than a few hours old. It is advisable to compare only those portions of chromatograms boiling above pentadecane in order to minimize the difference resulting from weathering. Reference oils can be weathered in the laboratory using the method of Reference 10.

Light distillate fuels cannot survive heavy weathering and have few hydrocarbons above C15. Comparison of the residues of these oils can only be done qualitatively - from about C8-C15.

d. A direct comparison of chromatograms will establish identity or nonidentity between samples. The comparison involves a peak-for- peak matching, noting differences or similarities in relative peak height. If the chromatograms are the same on the basis of the peak-for-peak matching, there is a high probability that the samples are from the same source. A mismatch occurs when the chromatograms are different. The differences may be due to the presence of one or more components or petroleum products in one sample relative to another. Spill samples may contain components such as cleaning detergents, plasticizer, paint vehicles, etc. The presence of one or two components in a spill sample which are absent in a suspect petroleum oil is not absolutely indicative of non-identity.

e. If the ratio of pristine/phytane in the unknown and reference petroleum oil match with $\pm$ 15%, the fingerprint match is confirmed.

11.5 Percent Recovery of Surrogate Standard (5)

Percent recovery is calculated using the 5-androstane GC internal standard, (C_{is}).

$$\% \text{ SS recovery} = \frac{(A_{ss})(C_{is}) \times 100}{(C_{ss})(A_{is})}$$

Where: C_{ss} = Concentration of surrogate

A_{ss} = Area of surrogate

SS = Surrogate

Percent recovery based on External Calibration

$$\% \text{ SS recovery} = \frac{C_{sf}}{C_{ss}} \times 100$$

Where: C_{sf} = Concentration of surrogate found

11.6 Percent Solids (P)

$$P = \frac{D_s}{T_s} \times 100$$

D_s = Weight dry Sample, g

T_s = Weight wet Sample, g

11.7 Dry Weight (S)

$$S = \frac{\text{Wet Weight} \times P}{100}$$

12.0 REPORTING REQUIREMENTS AND DELIVERABLES

The following minimal information must be provided to the Department on request. The Laboratory must keep this information on file and available for inspection by the Department as per N.J.A.C. 7:18 (16).

12.1 Chain of custody documents. For every sample submitted to the laboratory, both field and laboratory chain of custody documents MUST be provided at the end of the final data report. The chain of custody must show the signatures of the sample custodian, extraction supervisors and any other personnel who handled the sample. It must clearly track the movement of the sample through the laboratory by showing the relinquishing and acceptance of the sample by each person.

12.2 Methodology Review

The laboratory shall provide a brief narrative outlining the essential points of each method actually employed in the analysis of the samples submitted to the laboratory.

12.3 Non-Conformance Summary Report

The laboratory shall describe in narrative and/or tabular from any item which does not conform to the requirements of this method. This shall include but is not limited to a discussion of missed holding times, of failed Quality Assurance/Quality Control criteria, sample matrix effects on the analysis, sample dilutions, re-analyses, corrective actions taken and deviations from the analytical method specified on the analytical request form or the preparative methods permitted.

12.4 Sample Data Package - must contain the following information in sequence:

12.4.1 Quantitative Sample Results Summary (uncorrected for blank), Blank Results and Method Detection Limits.

12.4.2 Qualative Sample Result Summary (For fingerprint work)

12.4.3 Quantitation Reports

12.4.4 Sample Chromatograms

The chromatograms must be clearly labeled with the following information:

a. Sample identification number.
b. Volume injected.
c. Date and time of injection.
d. GC Column identification.
e. GC instrument identification - exact instrument employed.
f. Positively identified compounds, either directly above the peak or on a printout of retention times, if the retention times are printed on chromatograms.
g. Internal Standards labeled.

h. Surrogate labeled.
i. Analyst signature.

12.5 Quality Control Summary - must contain the following items:

a. Surrogate Recovery Summary
b. QC Check Sample Recovery
c. Method Blank Summary
d. Matrix Spike Summary
e. Duplicate Summary

12.6 Standard Data Packages - must contain the following items:

a. Initial Calibration Data Summary
b. Continuing Calibration Data Summary
c. Chromatograms of Standards and Quantitation Reports
d. Chromatograms of Reference Standards where applicable

12.7 Raw QC Data Package - additionally must contain the following items:

a. Blank Chromatograms
b. QC Check Sample Chromatograms
c. Blank spike Chromatograms.

12.8 Qualitative Sample Results Summary - List Sample Fingerprint matches (ASTM D3328(I)).

a. Based upon the visual comparison of source chromatograms, and after considering weathering, report the sample of unknown origin as belonging to one of the categories below:

1. Match - The chromatogram is identical to one, or more, of the samples submitted for comparison.

2. Probable Match - The chromatogram is similar to one, or more, of the samples submitted for comparison, except: (a) for changes which can be attributed to weathering, or (b) differences attributable to specific contamination.

3. Indeterminate - The chromatogram is similar to one, or more, of the samples submitted for comparison, except for certain differences as in 2, of such magnitude that it is impossible to determine whether the

unknown is the same petroleum oil heavily weathered, or a totally different oil.

4. Mismatch - Unlike the samples submitted for comparison.

b. Compare unknowns to a library of the products listed in Section 1.1.

13.0 REFERENCES

1. ASTM, "Comparison of Waterborne Petroleum Oils by Gas Chromatography", Method D3328-90, Annual Book of ASTM Standards, Volume 11.01, Philadelphia, PA, 1990.

2. Staff, Oil Spill Identification System, Report No. GCD-52-77, U.S. Coast Guard R & D Center, page Dl. National Technical Information Center, Springfield, VA, 1977.

3. Leaking Underground Fuel Tank, Task Force "Leaking Underground Fuel Tanks Field Manual," Appendix C, State Water Resources Control Board, Sacramento, CA 1988.

4. U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste – Physical Chemical Methods, Office of Solid Waste, Washington, DC, Publication SW846, 3rd ed. update III 1996.

5. Douglas, G.S., "Total Petroleum Hydrocarbons in water and Soil", Verbal Communication, Enseco Corp., Cambridge, MA 1988.

6. Miller, M., Analytical Chemistry Manual for Petroleum Products in the Environment, NJDEPE OQA-QAD-002, 1992.

7. U.S. Environmental Protection Agency, Federal Register, 40 CFR 136, Appendix A and B, Volume 49, No. 209, July 1, 1990.

8. "OSHA Safety and Health Standards, General Industry," (29CFR1910), occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

9. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

10. ASTM, "Practice for Preparation of Samples for Identification of Waterborne Oils," "Method 3326-90, Annual Book of ASTM Standards, Vol. 11.02, Phildelphia, PA 1982.

11. ASTM, "Practice for Identification of Waterborne Oils," "Method 3415-90, Annual Book of ASTM Standards, Vol. 11.02 Philadelphia, PA, 1992.

12. Zafiriou, 0., Stainken, D. et al, "Correlation of Oils and Oil Products by Gas Chromatography," Environmental Protection Technology Series, EPA-600/2-77-163, August 1977.

13. Potter, Thomas, "Fingerprinting Petroleum Products, Unleaded Gasolinell , Proceeding of the Fourth National Conference of Hydrocarbons Contaminated Soils, Chapter 8, page 83, Lewis Publishers, Chelsea, MI, 1990.

14. Remeta, D.P., and Grundfeld, M. "Emergency Response Analytical Methods for Use On Board Mobile Laboratories," Internal Communication, USEPA Hazardous Waste Engineering Research Laboratory Release Control Branch, 1987.

15. Dudenbostel, B.F., "Use of GC Peak Height Ratios for Passive Tagging of Petroleum Oils", Anal. Quality Control Newsletter, #18, July 1973.

16. NJDEPE, "Regulations Governing Laboratory Certification and Standards of Performance", N.J.A.C. 7:18 as amended July 1984.

17. Taylor, J.K. Quality Assurance of Chemical Measurements, Chapter 14, Page 129, Lewis Pub. Chelsea, MI, 1987.

18. Driscoll, J.D. , Ford,J. et.al, "Gas Chromatographic Detection and Identification of Aromatic and Aliphatic Hydrocarbons in Complex Mixtures by Coupling Photoionization and Flame Ionization Detectors",, J. Chromatography, 158, pp. 171-180, 1978.

19. Cox,R.D. and EarpR.F., "Determination of Trace Level Organics in Ambient Air by High resolution Gas Chromatography with simultaneous Photoionization and Flame Ionization Detection," Anal. Chem. 54 pp. 22652270, 1982.

20. Davis,K.V., "A New Photoionization/Flame Ionization Series Detector for Gas Chromatography," presentation, Pittsburgh Conference, March 1990.

21. "High Resolution Chromatography Products," J&W Scientific; Folsom, CA, p. 38, 1990.

22. "Petroleum/Chemical Applications Guide," Supelco Inc., Bellefonte, PA, 1990.

23. Thomas, D.H. and Delfino, LI., "A Gas Chromatographic/Chemical Indicator Approach to Assessing Ground Water contamination by Petroleum Products, " Ground Water Monitoring Research, p.90, fall, 1991.

24. Miller, M.W., Appleby, C. Wright,D., Skelton, P., "A gas Chromatography Method to Replace Method 418.1 for the Determination of Total Petroleum Hydrocabons", Submitted to Journal of Soil Contamination, Sept. 11,1998

25. Florida Department of Environmental Protection, "Method for Determination of Petroleum Range Organics," Method #FL-PRO, rev.1a,March 14,1997.

26. Schumacher, D.K., direct communication, "The Use of Chlorobenzene as a Low Temperatue Surrogate," Lancaster Laboratories, Inc., Lancaster, PA August 1998.

Figure 1 - Calibration Standard 50 ug/Kg (C8-C42)

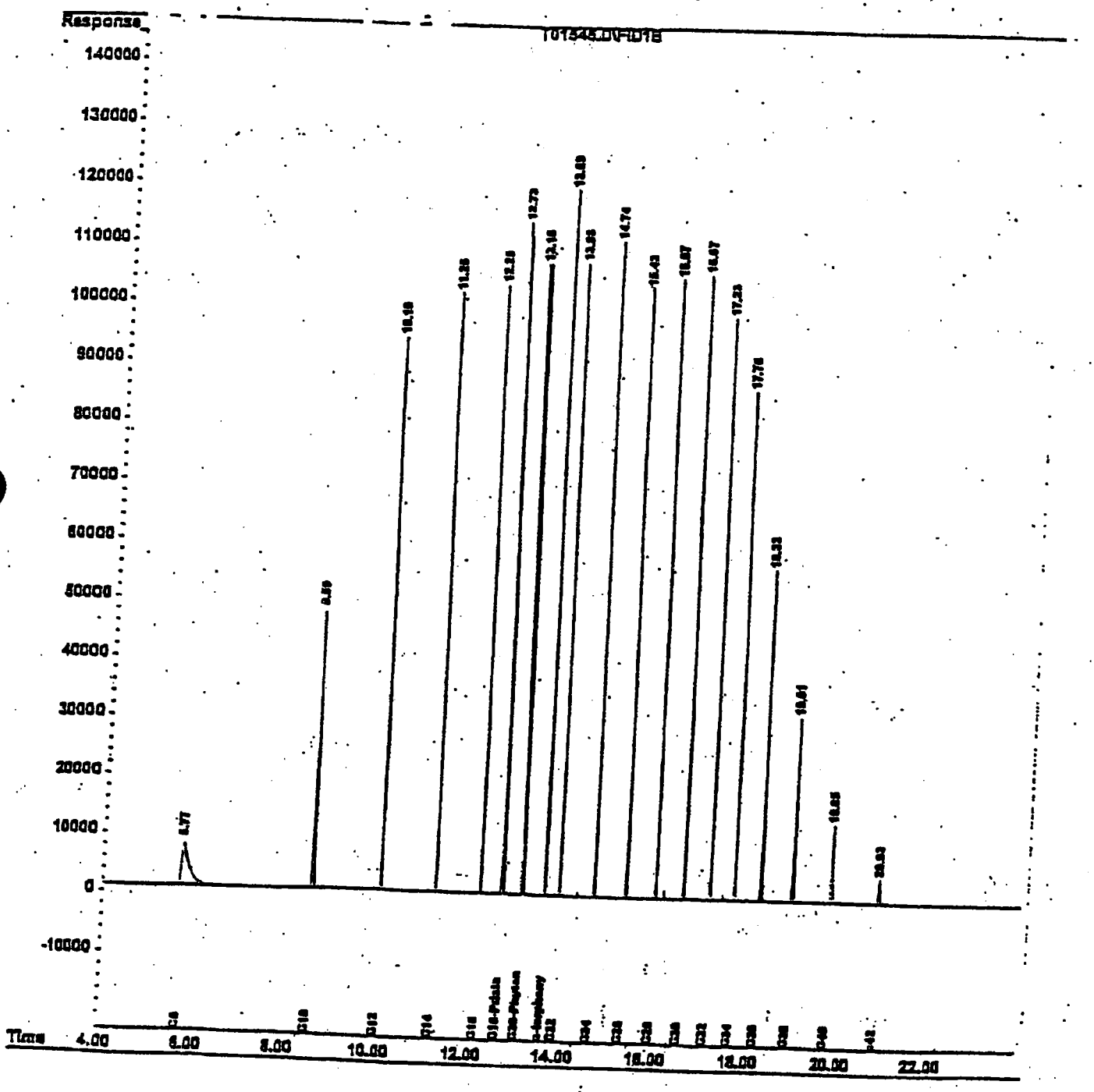


Figure 2 - MDL Study 250 ug/Kg in Soil

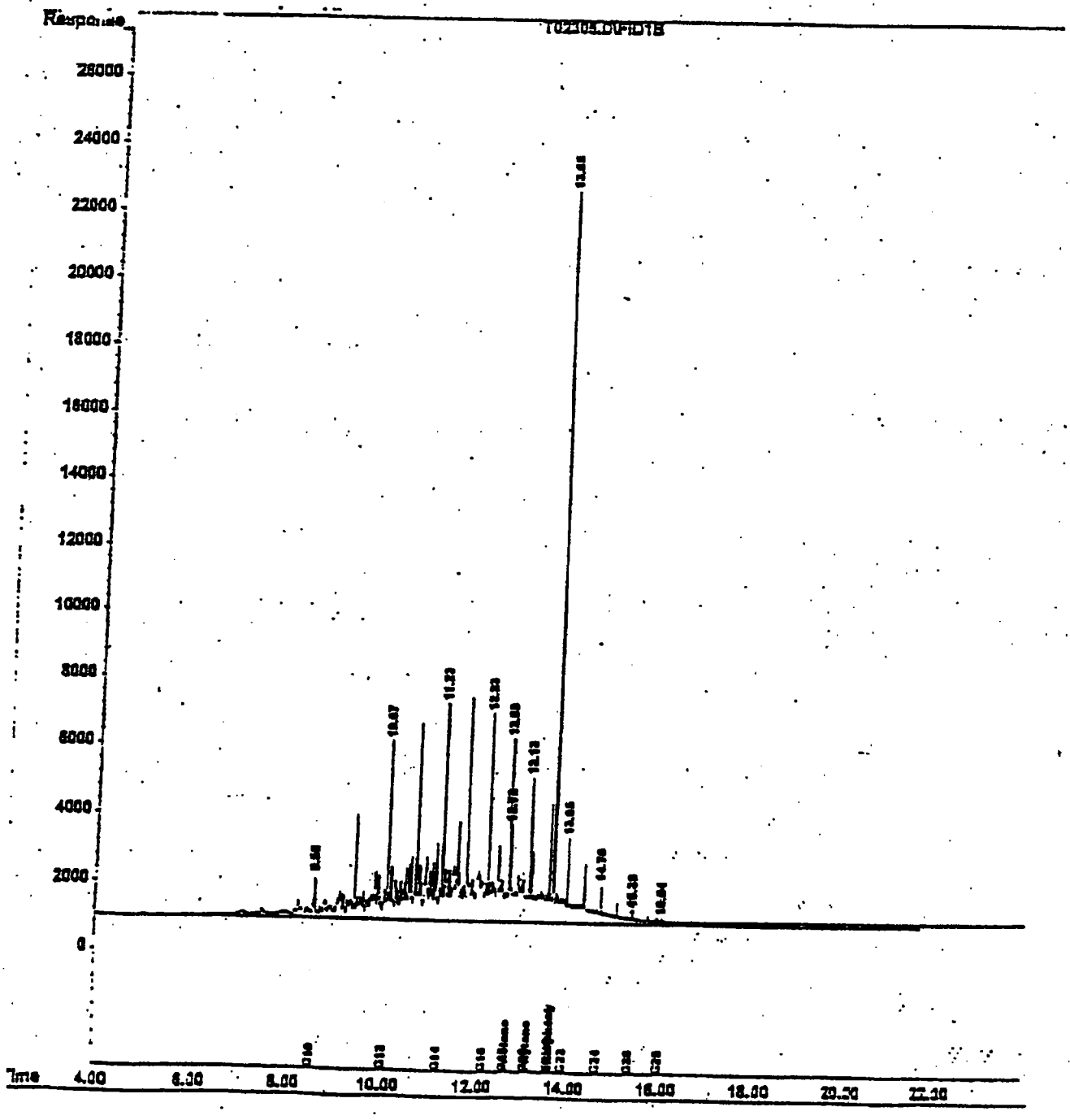


Figure 3 - Methylene Chloride Blank

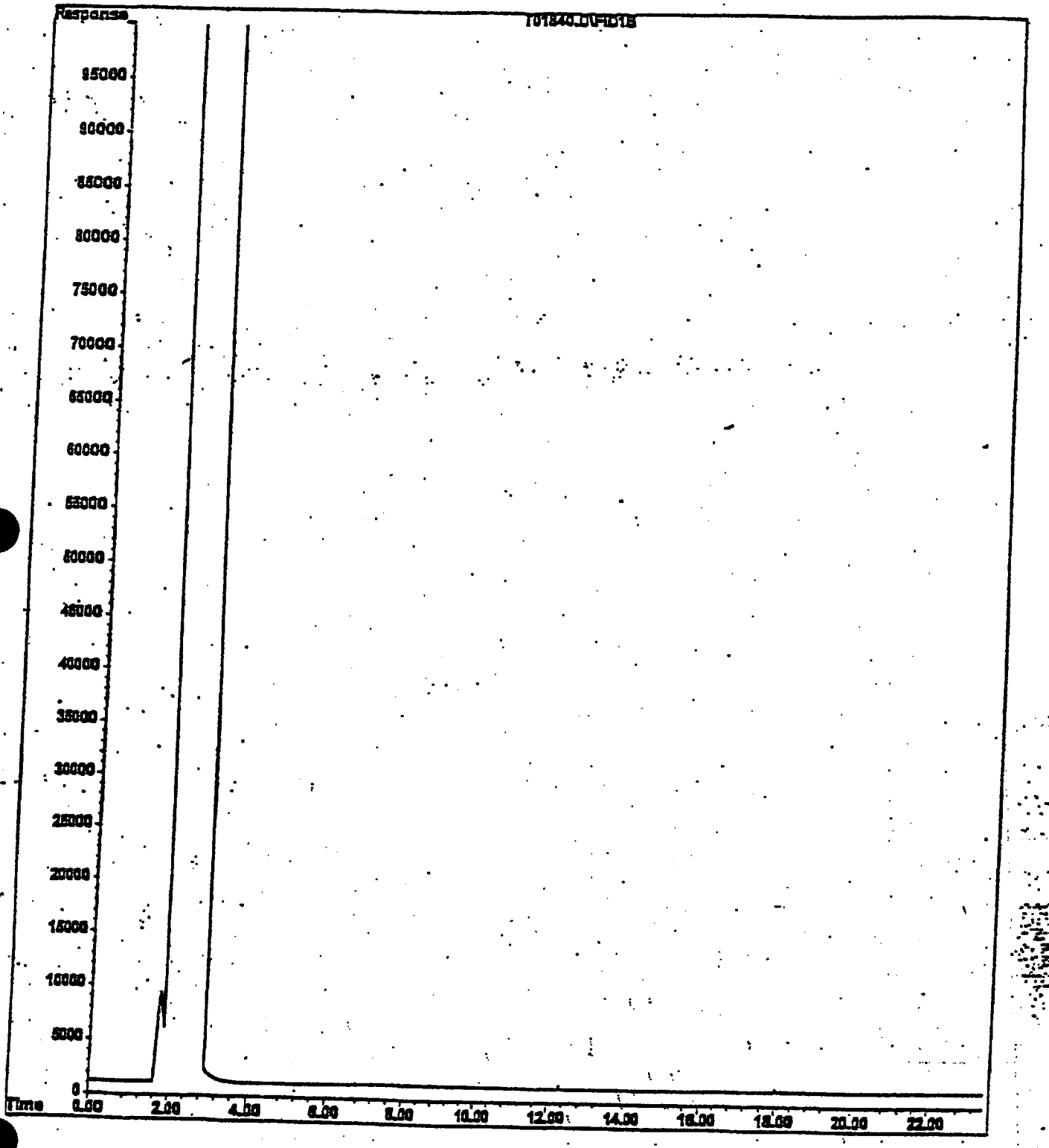


Figure 4 - Diesel Fuel #2 / Methylene Chloride Soxhlet Extraction